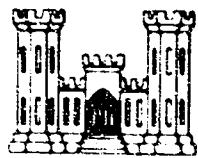


PRESSURE GROUTING FINE FISSURES

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PREFACE

The work reported herein was authorized by the Office, Chief of Engineers, as part of Item CW 612, "Prepakt Concrete and Grouting," of the Corps of Engineers Civil Works Investigations program, and completed under Item CW 550, "Grouting Research - Concrete Dam Foundations."

The investigation was begun in 1949 and was conducted by the Concrete Division of the Waterways Experiment Station under the supervision of Mr. V. L. Minear, formerly of the Office, Chief of Engineers, and Mr. H. K. Cook, formerly Chief of the Concrete Division. Personnel of the Waterways Experiment Station actively engaged in the work were T. B. Kennedy, C. H. Willetts, W. O. Crawley, R. L. Curry, B. Mather, K. Mather, and L. Pepper. This report was prepared by Mr. Cook and Mr. Kennedy.

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SUMMARY

This investigation of various grout mixtures and grouting pressures to determine their effects on the grouting of fine fissures was performed in three stages. In the first stage the lowest water-cement ratio grouts that could be pumped through cracks of 0.01-, 0.02-, and 0.03-in. thickness, formed between specially prepared concrete slabs, using 100-psi pressure were determined. Standard field grouting equipment and methods were used. The grouts investigated consisted of neat cement, cement plus fly ash, and cement plus fly ash plus Intrusion Aid. The second stage provided information on grout penetration and the quality of hardened grout films obtained at pumping pressures of 25 and 50 psi using the same grout combinations as in the first stage and an additional combination of cement plus Intrusion Aid, through crack thicknesses of 0.02 and 0.03 in. Tests were also made on neat-cement grout plus calcium lignosulfonate (RDA) at 25-, 50-, and 100-psi and on neat-cement grout plus Intrusion Aid at 100 psi. In the third stage information was obtained on the quality of hardened grout films and the penetration of 0.03-in. cracks using a 50-psi pumping pressure and grouts containing various proportions of cement, cement plus fly ash, cement plus RDA, cement plus fly ash plus RDA, cement plus slag, cement plus pumicite, and cement plus opaline shale. Tests were also made for consistency, bleeding, setting time, and solubility of the hardened grout films in distilled water.

The general conclusions and indications derived from the three stages of the investigation are contained in the following paragraphs.

The surface condition of the fissure, the ratio of the maximum grain size of the solids in the grout, the water-cement ratio of the grout and the pressure at which it is pumped all influence the width of crack that can be penetrated. The use of a fine sieve cloth to remove oversized particles from the grout seems warranted.

Poor bond between grout film and the top of a crack appeared to be normal because some bleeding almost inevitably occurs with grouts having water-cement ratios practicable to pump. Grout films with water-cement ratios of 0.5 or less bled little and were hard and dense. Setting time increased with increasing water content, and neat grout with a water-cement ratio of 3.8 by weight required ten days to set.

The use of fluidifiers allowed some reduction in water-cement ratio that could be forced into a given crack thickness. Their use increased setting time, decreased bleeding, and had little effect on the solubility of the grout film as judged by leaching tests.

An approximately straight-line drop in pressure occurred from the intake to the exhaust end of the crack when cracks of 0.02- and 0.03-in. thickness were grouted. With finer fissures the pressure drop was steeper for the first 12 in. than for the remaining 36 in. of crack length.

Frequently the solids appeared to have agglomerated in hardened grout films containing fly ash. The use of ground, water-quenched blast-furnace slag, pumicite, and opaline shale appeared to reduce bleeding and improve the appearance of hardened grout films.

Resistance of grout films to leaching increased with age and with decreasing water-cement ratio. Ground slag and opaline shale reduced the solubility of grout. The effects of pumicite and fly ash on solubility were inconclusive.

PRESSURE GROUTING FINE FISSURES

PART I: INTRODUCTION

Purpose of Investigation

1. It is recognized that the strength and impermeability of a grout film follow the water-cement ratio law as do mixtures of other materials containing water and cement. Because of this fact it is desirable, where important work is concerned, to use grouts containing as low water contents per unit volume as possible. Since the lower the water content the thicker the grout, it is also important to determine the thickest consistencies that will penetrate cracks of various widths at given pressures.

2. In sealing fine seams, grouts having water contents as high as 20 to 1 (20 cu ft of water per bag of cement) are sometimes used, and grouts of a consistency as thin as 10 to 1 are not uncommon. The assumption has been that thin grout placed under sufficient pressure to force out the excess water used to obtain such a consistency will form a hard durable film. Whether it is always possible to exert enough pressure to squeeze out this excess water is questionable, and for this reason a knowledge of the physical nature of the grout films produced by fairly high water-cement ratios would be valuable. Such grouts are believed to be extremely pervious and, in time, susceptible to leaching that will transform an impervious area into a pervious one.

3. The purpose of the program was to obtain information on the degree to which the penetration of fine fissures by grout was influenced by surface texture of specimen, pumping pressure, water-cement ratio, chemical fluidifiers, and finely divided mineral additives. It was also desired to determine the effect of these factors on the quality of the hardened grout films.

Description of Investigation

4. Preliminary tests were conducted to develop the best technique

for the grouting tests. It was found that the most practical approach to use in penetrating fine cracks is that used in the field wherein a very dilute grout is used first and is gradually thickened by addition of solid materials until refusal results. This procedure was adopted and enabled the operators to determine the stiffest grout, of any type tested, that could be pumped through the three fissure thicknesses selected for investigation at pressures up to 100 psi.

5. The study was conducted in three separate stages. The scope of each stage was as follows:

- a. The first stage furnished data on the lowest water-cement ratios of grouts that could be pumped through fissures of 0.01-, 0.02-, and 0.03-in. thickness at 100-psi pressure, using standard field equipment and methods, under the following grout conditions: (1) neat cement; (2) cement plus fly ash; (3) cement plus fly ash plus Intrusion Aid. A study of consistency, bleeding characteristics, and setting times of the various grouts was also made. After the grouting tests were completed, chemical and petrographic examinations of bleed water and solid residues from some of the high water-cement ratio grouts, and further bleeding tests were made. Results of these supplemental tests are contained in appendix A.
- b. The second stage provided data on grout penetration obtained at pumping pressures of 25 and 50 psi using the three grout conditions of the first stage and the additional condition of cement plus Intrusion Aid. Tests were also made of neat-cement grout plus calcium lignosulfonate (RDA) at 25, 50, and 100 psi, and of neat-cement grout plus Intrusion Aid at 100 psi.
- c. The third stage consisted of: pumping tests through a 0.03-in.-thick crack at 50 psi using the seven grout conditions listed below; tests of the grouts themselves for consistency, bleeding, setting time; and tests to determine the apparent quality of hardened grout films and their solubility in distilled water under various leaching conditions.

Grout Conditions

(1) Neat cement	(5) Cement plus slag
(2) Cement plus fly ash	(6) Cement plus pumicite
(3) Cement plus RDA	(7) Cement plus opaline shale
(4) Cement plus fly ash plus RDA	

Some of the above materials were used in various proportions of one to the other so that a total of 17 different

grout mixtures were pumped. These are described in detail later in the report. Stage three also included detailed tests of consistency, bleeding, and setting time of the fluid grouts, and leaching tests made with distilled water on the hardened grouts as well as examinations of the hardened grout films for apparent quality. These bleeding tests and the examination of the hardened grout films were in addition to the work described in appendix A and done in the first stage of the investigation.

6. This report contains the essential information obtained in the three stages. This information has been grouped so that all data bearing on one condition are presented together rather than discussed under the separate stages.

PART II: MATERIALS, EQUIPMENT, AND SPECIMENS

MaterialsCement

7. Type II cement was used in all three stages of the investigation and was obtained from the same mill but in three different shipments. The chemical and physical data for each shipment, designated by the numbers RC-183, RC-186, and RC-233, are shown below:

	Stage 1 (RC-183)	Stage 2 (RC-186)	Stage 3 (RC-233)
<u>Chemical Properties</u>			
Constituents, %			
SiO ₂	22.8	22.5	22.2
Al ₂ O ₃	4.3	5.3	4.8
Fe ₂ O ₃	3.8	3.2	3.7
CaO	63.2	64.0	63.9
MgO	3.3	3.0	3.1
SO ₃	1.5	1.5	1.5
Ignition loss	0.55	0.68	0.52
Insoluble residue	0.24	0.18	0.19
Na ₂ O	0.17	0.21	0.13
K ₂ O	0.48	0.34	0.41
Total alkalies as Na ₂ O	0.49	0.44	0.40
Calculated Compounds, %			
C ₃ S	45	46	50
C ₂ S	32	30	26
C ₃ A	5	9	6
C ₄ AF	12	10	11
CaSO ₄	3	3	3
<u>Physical Properties</u>			
Normal consistency	25.2	24.2	25.0
Setting time, Gilmore, hr-min:			
Initial	4:00	3:25	3:15
Final	6:20	6:25	4:15

(Continued)

	Stage 1 (RC-183)	Stage 2 (RC-186)	Stage 3 (RC-233)
<u>Physical Properties (Continued)</u>			
Autoclave expansion, %	0.09	0.07	0.10
Air content of mortar, %	4.9	7.3	3.5
Compressive strength of mortar, psi:			
3 days	1540	1260	1625
7 days	2390	2010	2875
28 days	4025	3300	5085
Fineness, cm^2/g :			
Wagner	1805	1720	1840
Blaine	3170	2965	3080
Sieve analysis, dry:			
No. 50 (% retained)	0.2	0.0	0.0
No. 100 (% retained)	0.1	0.1	0.0
No. 200 (% retained)	2.4	4.3	1.3

Fly ash

8. The fly ash used in all three stages came from the Chicago region but in different shipments. A chemical analysis was not made of the fly ash used in stages 1 and 2. The chemical analysis of the fly ash used in stage 3 is shown in paragraph 9. The physical characteristics of the fly ash used in the respective stages are listed in the following tabulation:

Sieve No.	Opening in.	Sieve Analysis, Dry			
		Per Cent Retained	Stage 1	Stage 2	Stage 3
50	0.0117	0.4	0.2	0.9	
100	0.0059	1.3	1.1	0.9	
200	0.0029	6.2	6.0	2.5	
Fineness, cm^2/g :					
Blaine		2795	2795	----	
Fisher		----	----	3335	
Specific gravity		2.43	2.43	2.48	

Other mineral admixtures

9. The following mineral admixtures were used in stage 3 only:

- a. Slag, water-quenched, ground, laboratory No. RC-216.
- b. Pumicite, Friant, laboratory No. AD-6.
- c. Opaline shale, calcined, Napa, laboratory No. AD-13.

Data on these materials and on the fly ash used in stage 3 are listed on the following page:

	Fly Ash AD-3 %	Slag RC-216 %	Pumicite AD-6 %	Opaline Shale AD-13 %
<u>Chemical Properties</u>				
<u>Constituents, %</u>				
Moisture loss	0.20	-----	0.83	1.2
SiO ₂	47.2	35.8	68.8	70.1
Al ₂ O ₃	19.5	15.8	14.8	19.3
Fe ₂ O ₃	18.2	1.2	1.4	5.9
Mn ₂ O ₃	0.07	0.62	0.05	0.05
P ₂ O ₅	0.23	-----	0.02	0.14
CaO	5.3	35.5	0.65	0.22
MgO	1.2	10.4	0.33	0.85
Sulfide sulfur	0.05	-----	0.00	0.00
SO ₃	2.2	0.1	0.03	0.10
Ignition loss	0.8	0.23	3.9	1.26
Insoluble residue	70.4	0.52	94.6	86.9
Na ₂ O (gravimetric)	0.69	-----	-----	-----
K ₂ O (gravimetric)	1.16	-----	-----	-----
Total Na ₂ O (gravimetric)	1.45	-----	-----	-----
Na ₂ O (flame)	1.62	0.11	1.38	0.40
K ₂ O (flame)	1.98	0.65	4.96	0.61
Total Na ₂ O (flame)	2.92	0.54	4.64	0.80
CHCl ₃ sol	-----	-----	-----	-----
Total carbon	0.43	0.54	0.02	0.07
<u>Physical Properties</u>				
<u>Fineness, cm²/g:</u>				
Wagner	-----	3,430	-----	-----
Blaine	-----	5,795	-----	-----
Fisher	3,35	5,470	4,640	15,020
Specific gravity	2.48	2.85	2.35	2.35
<u>Sieve No., and sieve opening (in.)</u>				
50	0.0117	0.9	0.8	0.0
100	0.0059	0.9	0.4	0.4
200	0.0029	2.5	1.3	13.9

Chemical admixtures

10. Intrusion Aid from the same shipment was used in stages 1 and 2 of the program and was furnished by the Prepackt Concrete Company, Cleveland, Ohio. It causes slight expansion of the grout and is purported to prevent agglomeration of the solids and aid intrusion of the grout.

11. Calcium lignosulfonate, RDA, was used in stages 2 and 3 and was furnished by Dewey and Almy Chemical Company, Cambridge, Mass. Marasperse C was used in stage 2 only and was furnished by the Marathon Corporation, Rothschild, Wis. These materials were added for the same purposes as the Intrusion Aid, except that they do not have the expansion characteristics of the Intrusion Aid.

Grouting Equipment

12. The grout pump was a single-cylinder, reciprocating 3-3/4- by 2-1/2- by 5-in. steam pump with rubber piston and valves, air-driven for these tests. The grout mixer was a paddle-type machine powered by a motor from an electric drill. A photograph of the grouting assembly, including a typical specimen, as used in stage 1 of the program is shown on fig. 1. An important change in the equipment setup was made at the conclusion of stage 1 and was used throughout the remainder of the program. This change is shown schematically on fig. 2 and consisted of connecting the delivery line and a return line with a corresponding control valve immediately adjacent to gage A (gage 0 on fig. 2). It was suspected that the velocity of flow in the riser of the delivery line would be so low in some cases as to allow settlement of the coarser particles and thus result in a variable grout passing through the fissure. This supposition was confirmed by observing the flow of grout through a vertical lucite tube. This difficulty was largely overcome by opening the valve on the return line, as shown on fig. 2, sufficiently to insure a relatively constant rate of grout flow through the riser under all grouting conditions. In addition, three additional gages were installed along the specimen so that variations in pressure gradient could be determined during grouting operations.

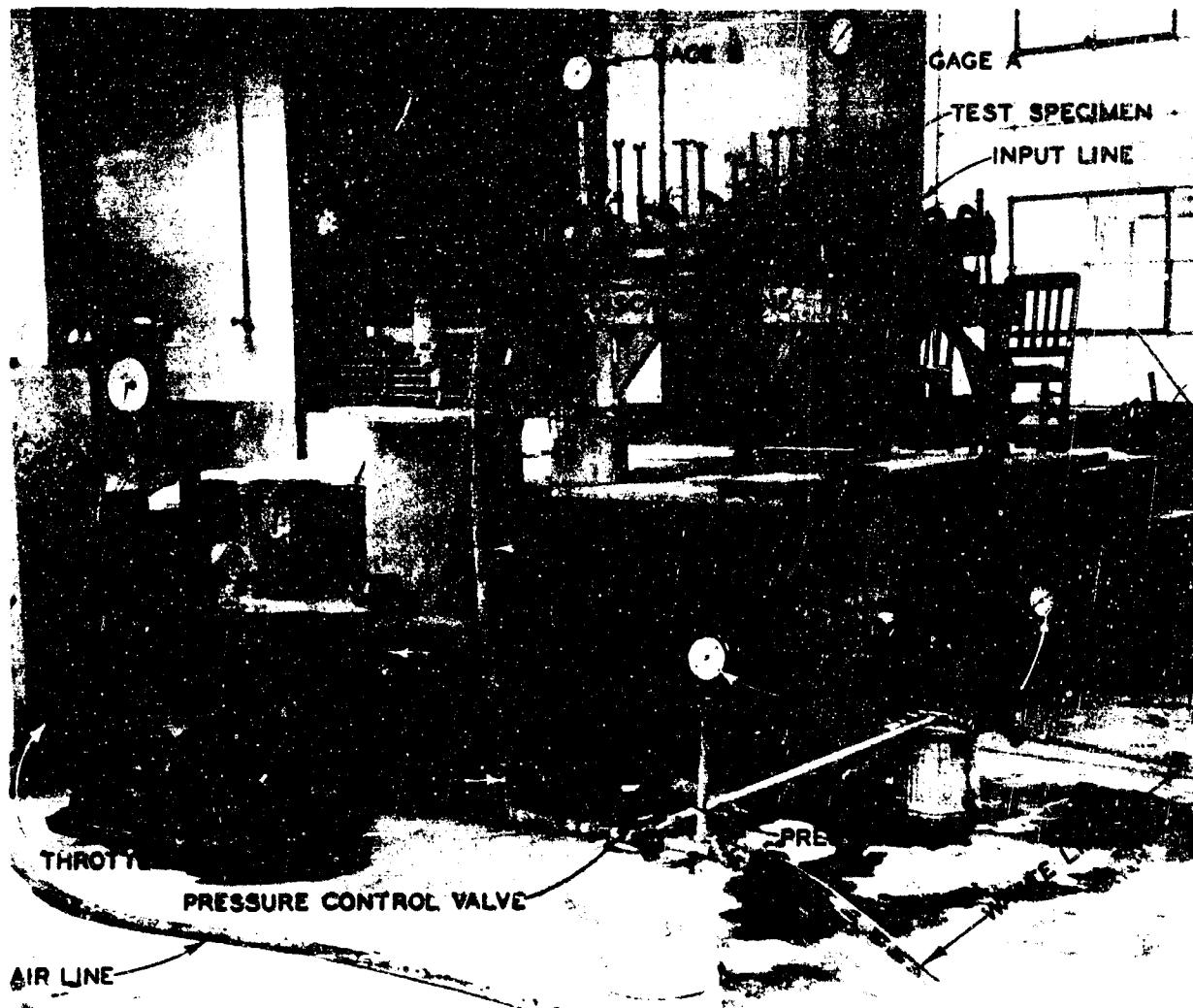


Fig. 1. Equipment setup for stage 1 grouting tests

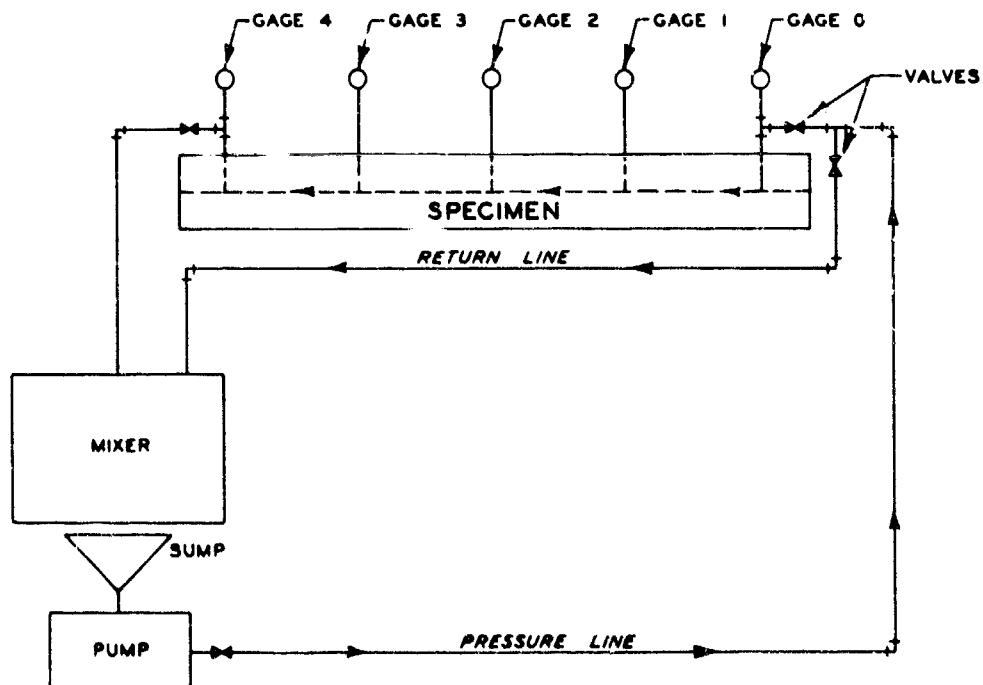


Fig. 2. Schematic diagram (not to scale) of grouting equipment setup for test stages 2 and 3

Specimens

13. It had originally been intended to cast prisms of high-strength concrete 6 in. wide by 7 in. deep by 52 in. long over-all, containing two 3/4-in. pipe nipples 48 in. apart embedded in the same side along the center line, with the axis of the nipple normal to the side surface and extending to a point just past the center of the prism. After curing for approximately seven days, the prisms were to be sawed from end to end, producing two pieces approximately 3 in. thick by 7 in. wide by 52 in. long with the saw cutting off the tip ends of the embedded pipe nipples. Then 1/2-in.-wide shimstock of the desired thickness was to have been placed between the two halves of the prism to serve as separator gaskets around the edges and ends and the halves fitted back together and held in place with C-clamps, thus forming a fissure 6 in. wide by 48 in. long between pipes. It was then planned to pump the grout into the pipe nipple at one end and out the pipe nipple at the other end.

14. This scheme did not prove successful because it was found impossible to make a perfectly straight cut with the diamond saw. Furthermore, the kerf of the saw was quite pronounced leaving a series of ridges and grooves which made a tight seal and control of the crack thickness impossible. Consequently, the specimens were cast in two pieces using a sheet of 1/4-in. plate glass as a separator in the plane where the saw cut would have been made. Several pieces of glass were measured for variation in thickness and none were found to vary over 0.002 in. Surfaces cast against the glass were smooth and true. The specimens for stage 2 and 3 were cast with five 3/4-in. pipe nipples, spaced 12 in. apart, along the center line of one slab to accommodate pressure gages and to allow flow of the grout. The specimens for stage 1 contained only two pipe nipples. The specimens were pumped in a horizontal position for the following reasons:

- a. The most dangerous conditions in grouting under a dam are caused by extensive open passages such as horizontal bedding planes. It was desired to learn something about grout flow and pressure distribution under such conditions.
- b. It was hoped to learn something concerning the validity of

the assumption that excess mixing water can be forced out of the grout by the application of pressure. It was believed that if this did occur a good bond would be made with the top as well as the bottom half of the specimen; if not, bleed water would prevent bond with the top half.

PART III: GROUT PUMPING TESTS

General ProceduresTreatment of grout

15. Dry materials were all sieved through a 30-mesh sieve and the mixed grout was passed through a 30-, 50-, or 100-mesh sieve depending upon the water-cement ratio. The thinnest grouts were passed through a 100-mesh sieve, thicker grouts through a 50-mesh sieve, and the thickest grouts through a 30-mesh sieve.

Grout injection

16. Grout injection was started by pumping water through the specimen followed by a thin grout which was gradually thickened by the addition of solids. The desired pressure was maintained on the specimen by operating the bypass valve so that only a controlled portion of the total flow of grout was shunted through the specimen. The pump was operated at a speed of approximately 72 strokes per minute for the major portion of the tests. At the lowest water-cement ratio for a given test more than one hour of pumping was usually required to discharge one cubic foot of grout through the fissure of a specimen. Grout flow stopped at any given pressure when the grout became ~~too~~ thick. In the stage 1 operations, pressure was maintained on specimens for 10 to 15 min after all flow had stopped with the valve at B (fig. 1) open at all times. Water continued to drip very slowly from the discharge line during final application of pressure, being squeezed out after separating from the grout film in the specimen. When gage B assembly was removed the short pipe nipple cast in the test specimen was invariably found to be full of hardened grout into which a pencil could only be forced with difficulty. This was the case regardless of the consistency of the grout being pumped at the end of the test. In stage 2 operations the valves at the entrance and discharge ends of the specimen were closed as soon as all flow had stopped and the pressure within the specimen was allowed to equalize insofar as internal conditions would permit. Pumping for stage 3 was in most instances conducted as in stage 1 although for some tests the valves were closed on both ends of the specimens as in stage 2.

Test conditions

17. The major portion of the tests on the various grout mixtures was performed at pressures of 100, 50, and 25 psi; the grouts were pumped through cracks of 0.01-, 0.02-, and 0.03-in. thickness. Some of the tests, particularly those in stage 3 where the effects of the addition of several finely divided materials were investigated, were conducted only at a pumping pressure of 50 psi. The test conditions and the composition of the grout mixtures investigated in all three stages of the program are shown in table 1.

18. Observations were made during and after pumping for:

- a. The effect of specimen surface on the water-cement ratio and pumpability of the grout (stage 1 only).
- b. Lowest water-cement ratio of grout that would penetrate the three different fissure thicknesses.
- c. Pressure drop along the specimen.
- d. Rate of grout flow at a given pressure, water-cement ratio, and crack thickness.
- e. Relationship between pumping pressure and lowest water-cement ratio grout that a fissure would take.
- f. Relationship of particle size of grout solids to the width of crack that could be grouted.
- g. Effect of chemicals (Intrusion Aid and RDA) on the penetration qualities of grouts.
- h. Quality of hardened grout films, from visual observation.
- i. Strength of grout film as judged by shear tests (for stage 2 only).
- j. Special observations and tests were made in stage 3 for consistency of the grout at each change in water-cement ratio by measuring change in unit weight of the grout and the torque imparted to a piano-wire consistency meter.

Discussion of Pumping Tests

19. Table 1 shows the major results of the pumping tests for all combinations of materials tested. In most instances the lowest water-cement ratio shown for a given grout mixture and test condition was the lowest water-cement ratio that could be pumped. It is considered, however,

that the water-cement ratio at which the flow equals 0.1 cu ft per min (6 cu ft per hr) is the stiffest grout practical for field pumping.

20. Throughout this report water-cement ratio has been expressed by weight of water to cement or to cement plus other solids when materials such as fly ash were added to the grout.

Effect of surface texture of specimen on grout penetration

21. Pumping tests 2 through 12 were made using specimen surfaces from which the glaze, due to casting against the glass, was removed by light rubbing with a carborundum stone. Tests 13 through the end of the program were made using specimen surfaces from which the glaze had not been removed. The effects of type of specimen surface, fineness (maximum grain size) of the grout, and crack thickness on the water-cement ratio of the grout are shown on fig. 3 and discussed on the following page:

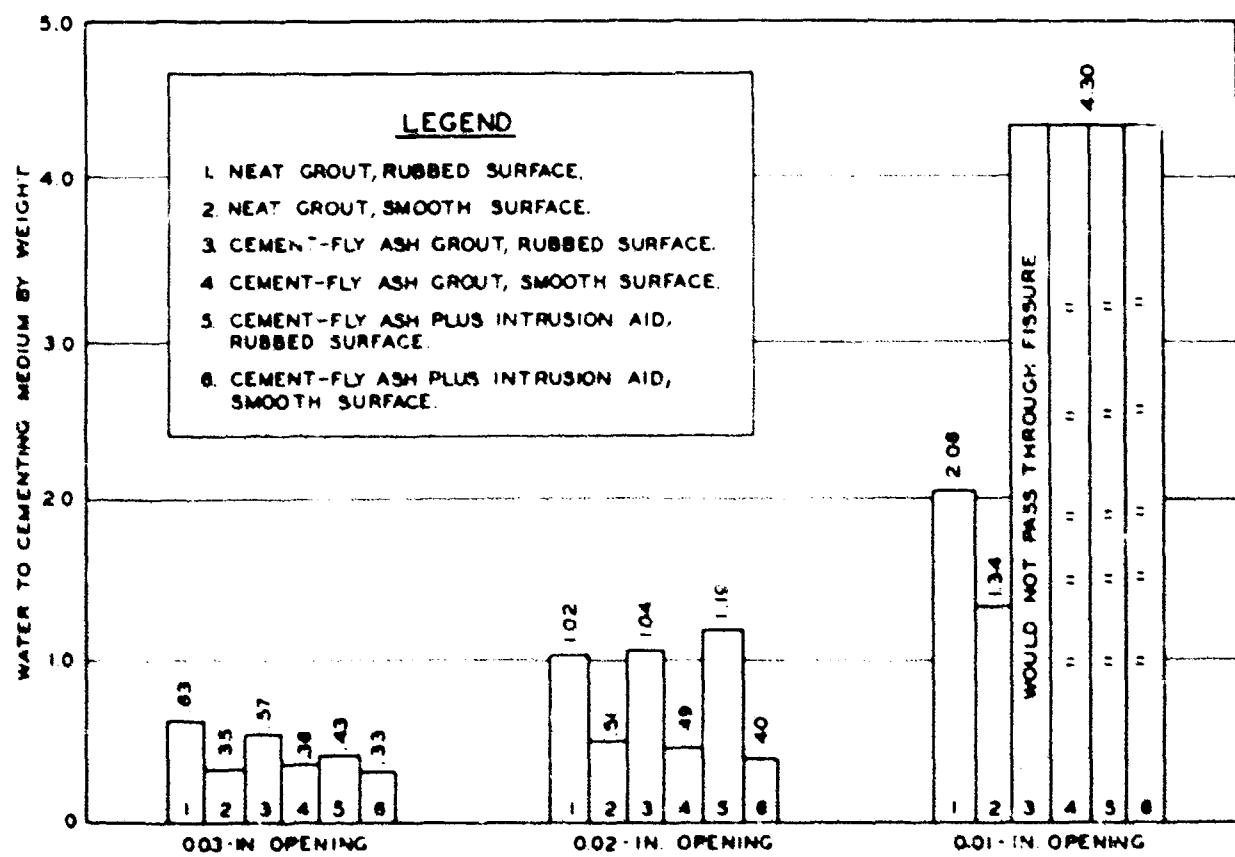


Fig. 3. Effect of crack thickness and surface texture, and grout fineness on water-cement ratio

- a. The combination of smooth surfaces and grout strained through a No. 50 sieve permitted the use of lower water-cement ratios for the 0.03- and 0.02-in. fissures than for the combination of rubbed surfaces and grout strained through a No. 30 sieve, for both neat grout and grout containing fly ash.
- b. The combination of smooth surfaces and grout strained through a No. 50 sieve permitted the passing of neat grout with a water-cement ratio of 1.34 by weight through the 0.01-in. opening as contrasted with a water-cement ratio of 2.06 with the rubbed surfaces and neat grout strained through a No. 30 sieve.
- c. Grout containing fly ash could not be pumped through the 0.01-in. opening regardless of the type of surface or when strained through the No. 50 sieve even with a water-cement ratio of 4.30, the highest water-cement ratio used in the tests. The reason for the poor pumpability of the fly ash grout is discussed in paragraph 22.
- d. The use of Intrusion Aid permitted a slight reduction in water-cement ratio when grouting the 0.03-in. fissures with rubbed surfaces as compared to the grout containing fly ash and no Intrusion Aid, but did not help when grouting the 0.02- and 0.01-in. fissures with rubbed surfaces.
- e. The use of Intrusion Aid permitted a slight reduction in water-cement ratio when grouting the smooth-surfaced 0.02- and 0.03-in. fissures but was ineffective in promoting penetration of the 0.01-in. fissures.

22. The physical data on the materials used in stage 1 showed the cement to have a greater specific surface (Blaine 3172 cm^2 per g, 97.3 per cent passing the No. 200 sieve) than the fly ash (Blaine 2795 cm^2 per g, 92.1 per cent passing the No. 200 sieve), and showed that the fly ash contained somewhat more than 0.4 per cent of material larger than 0.0117 in. (No. 50 sieve). This grain size exceeded the 0.01-in. opening of the smallest fissure and caused a grid of oversize grains to build up quickly during pumping which blocked the passage of the grout through the fissure regardless of whether or not the grout contained Intrusion Aid. Test 22 seemed to be an exception to the above but since a bad leak developed under the shim of this specimen during the test it is believed that the crack thickness was actually greater than 0.01 in., thus vitiating the results.

Influence of crack thickness on grout penetration

23. Fig. 4 is a bar graph showing the water-cement ratio for each type of grout at the stage where it just failed to penetrate the three fissure widths at the pumping pressures used. Grouts of only slightly higher water-cement ratios than those indicated penetrated the fissures. Examination of the figure shows that for neat-cement grout pumped at 25 psi the lowest water-cement ratio that would penetrate an 0.01-in. crack exceeded 2.67, when this pressure was increased to 50 psi the water-cement ratio exceeded 1.33 but an increase to 100 psi did not permit any further reduction in water-cement ratio.

24. Little practical difference was noted between the water-cement ratios of the neat grouts that would penetrate the 0.02- and 0.03-in.

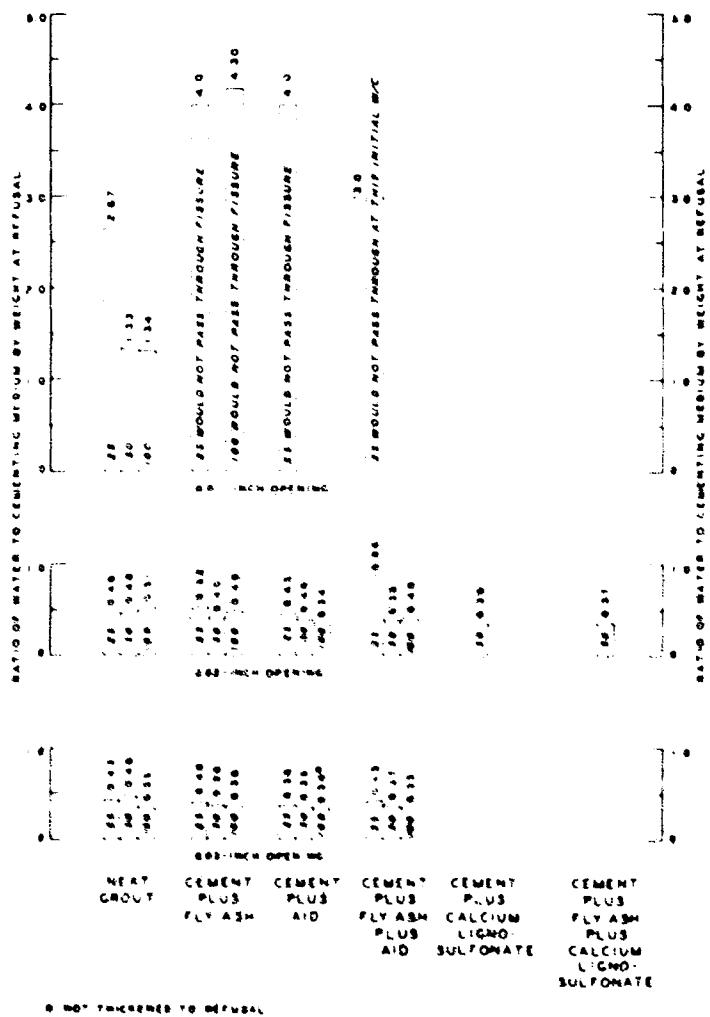


Fig. 4. Minimum water-cement ratio at refusal as influenced by crack thickness

cracks. Pumping pressure, within the range investigated, also had little effect on the ability of grouts of various water-cement ratios to penetrate the 0.02- and 0.03-in. cracks.

25. Grouting of any of the 0.01-in. cracks with grouts containing fly ash was found impracticable, regardless of the pressure applied, because of the maximum grain size of the fly ash.

26. The use of fly ash was of no benefit in grouting the 0.02-in. crack, since it permitted no practical reduction in water-cement ratio over neat grout. Pumping pressure, within the range used, was of little value in improving the penetration of the 0.02-in. crack with a cement and fly ash grout.

27. Grouts of cement and fly ash that would penetrate the 0.03-in. crack were of slightly lower water-cement ratio than the neat grouts that would penetrate cracks of similar width at 25 and 50 psi, but practically the same at 100 psi.

28. Use of Intrusion Aid in neat-cement grout permitted a slight reduction in water-cement ratio of the grouts that would penetrate both the 0.02- and 0.03-in. cracks at the pressures tested.

29. RDA was used only at 50-psi pressure on the 0.02-in. crack with neat cement and with cement plus fly ash; it appears to be as effective as Intrusion Aid with respect to grout penetration.

30. In the above discussion the effect of maximum grain size, as indicated in paragraph 22, should be kept in mind as well as the fact that fig. 4 indicates water-cement ratios at refusal, whereas a rate of flow of 6 cu ft per hr is the stiffest grout practical for field pumping.

Pressure drop along the specimen

31. The pressure gradient for the 0.02- and 0.03-in. cracks during pumping was approximately a straight line with zero pressure at the exit end of the specimen (gage 4), maximum pressure at the entrance end of the specimen (gage 0), and intermediate pressures at gages 1, 2, and 3. The static pressure, after pumping was stopped when the grout was thick (water-cement ratio of less than 0.5) and valves at both entrance and exit ends were closed, did not always equalize from end to end. This

may have been due to a thixotropic* condition developing in the thick grout immediately upon cessation of movement, and causing a blocking of the specimen between gages. For the 0.01-in. crack, the pressure drop during pumping did not follow a straight line. Fig. 5 is a series of curves of pressure gradients that developed when neat grouts were pumped at various water-cement ratios through a 0.01-in. crack at 25 and 50 psi. It will be noted that the gradient was fairly uniform along the whole specimen length while water was being circulated through it; however, as the grout was thickened flow diminished and the pressure gradient between the first two gages (first 12 in. of length) increased. This would apparently indicate that in the very fine seams as the grout thickened and the flow diminished the hydraulic pressure fell off rapidly with distance

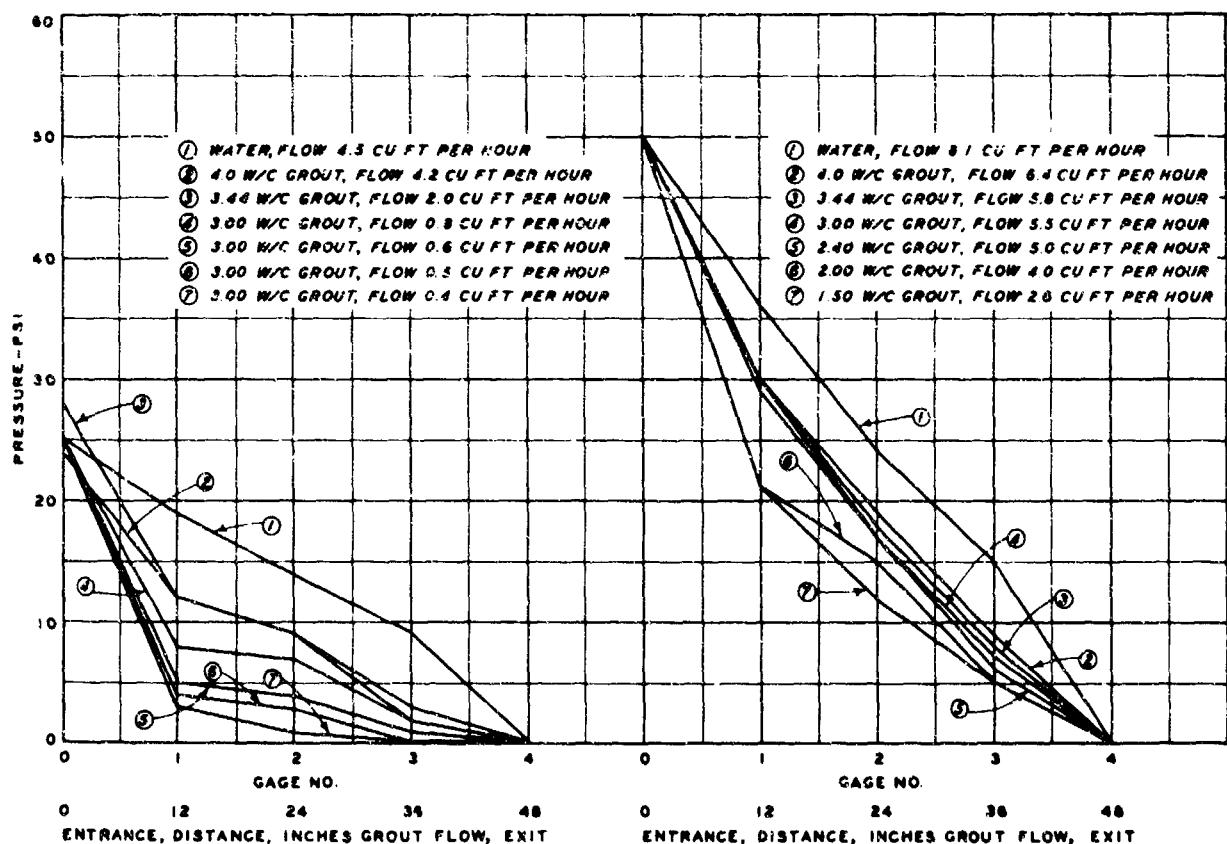
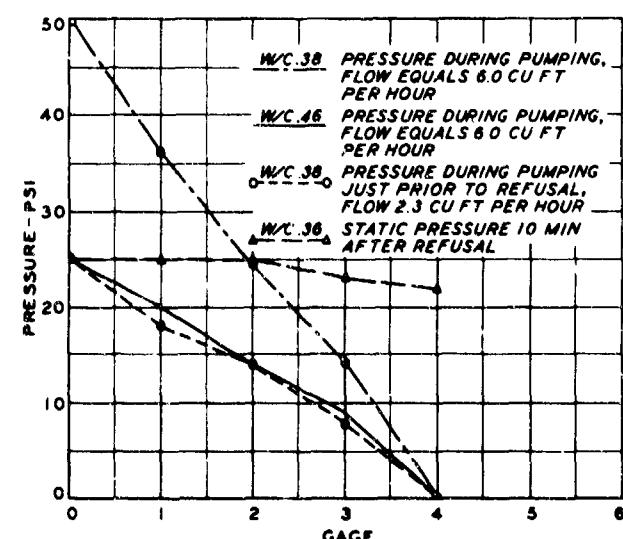
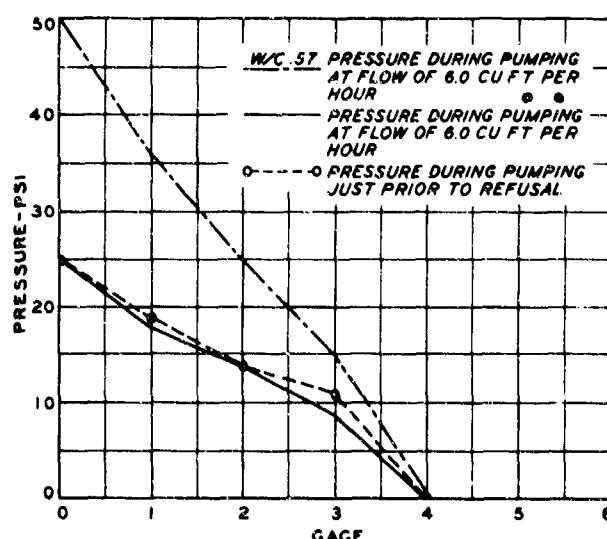
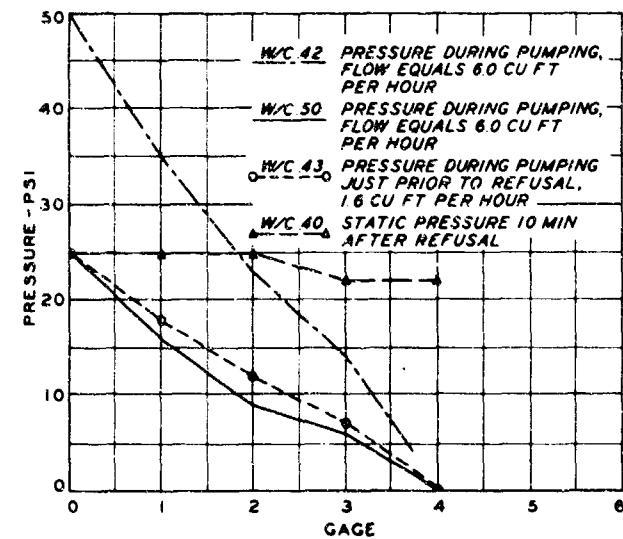
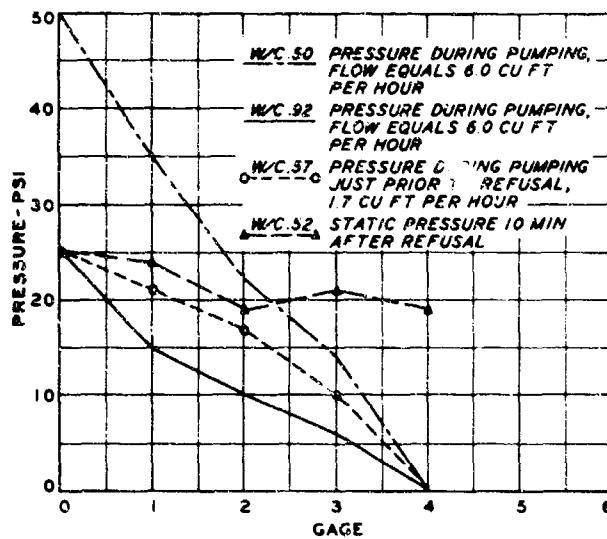
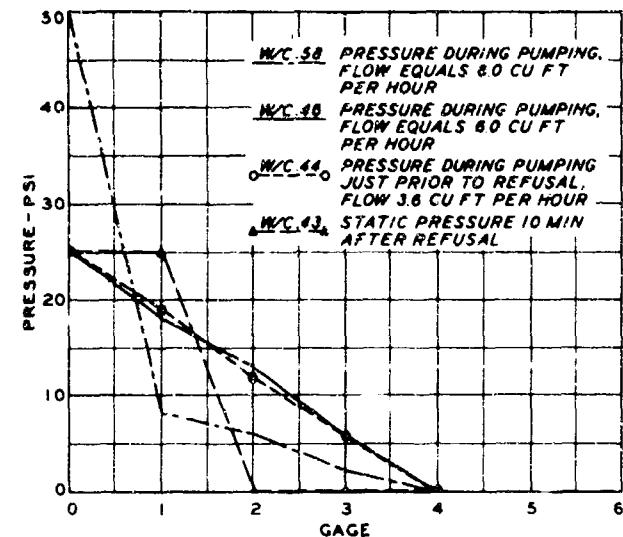
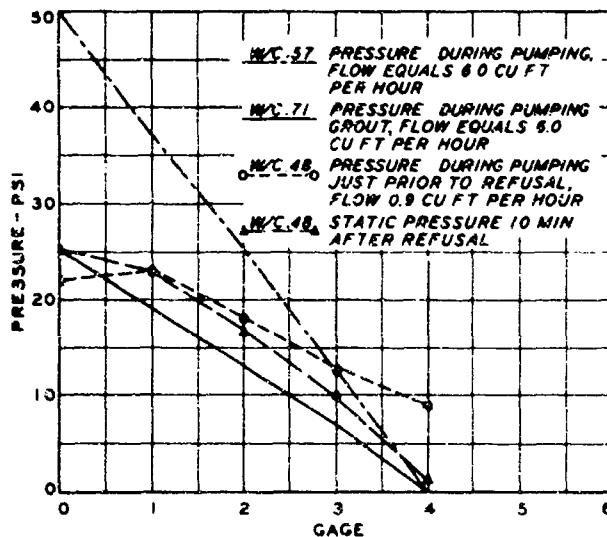


Fig. 5. Pressure gradients during grouting of 0.01-in. crack with neat grout

* A property sometimes exhibited by liquids of becoming gel-like on standing and liquid again on agitation.



from the intrusion point. Conditions of pressure gradient and static pressure after pumping are shown on figs. 6-14 for crack thicknesses of 0.02 and 0.03 in.

32. These figures show that in cracks with smooth walls and thicknesses of 0.02 in. or more the conditions of flow and pressure are similar to those of water. Irregularities in the curves are due to faulty operation of the gages occasioned by plugging. Water-cement ratio values at which the grouts would act like water, relative to pressure distribution, might be different in nature where fissure walls are rough.

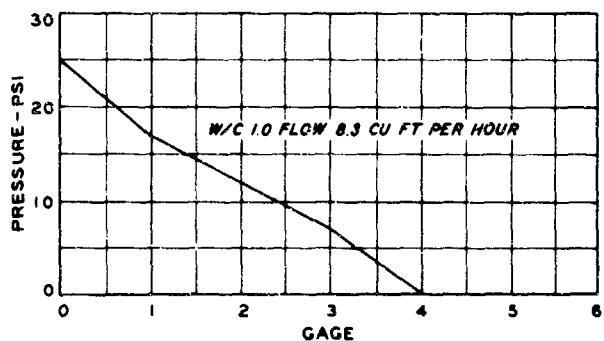


Fig. 12. Pressure gradient during grouting 0.02-in. crack, 1 cement:
1 fly ash + Intrusion Aid grout

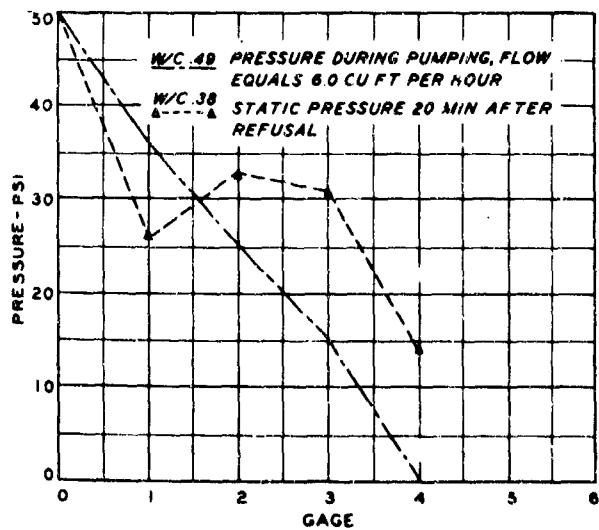


Fig. 13. Pressure gradient during grouting 0.03-in. crack, 1 cement:
1 fly ash + Intrusion Aid grout

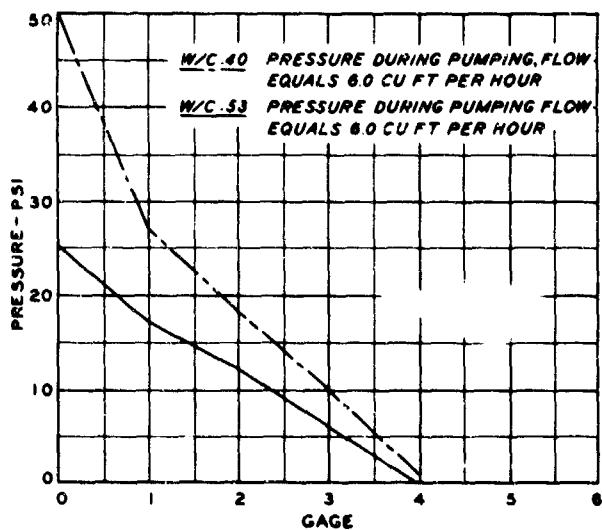


Fig. 14. Pressure gradient during and after grouting 0.02-in. crack,
1 cement:1 fly ash + Intrusion Aid
grout

Rate of flow at given pressure, water-cement ratio and crack thickness

33. Fig. 15 is a bar graph showing the amount (in cubic feet per hour) of various grouts, all with a water-cement ratio of 0.5, that could be pumped through 0.02- and 0.03-in. cracks at the three pumping pressures employed. Fig. 16 presents the same data in curves, omitting the data on the 1 cement:1 fly ash plus Intrusion Aid grout, which are anomalous and inconclusive.

34. Increasing pressure caused proportionately increasing flow. Apparently the flow of the 1 cement:1 fly ash grout was greater at equivalent pressures and crack openings than that of neat grout.

35. The use of Intrusion Aid in the neat grout increased flow at equivalent pressures and crack thicknesses over plain neat grout and caused greater flow than did the use of fly ash. Data on the effect of Intrusion Aid on the 1 cement:1 fly ash grout were inconclusive.

36. The rate of flow provides a realistic measure of the viscosity of a grout. Figs. 15 and 16 show that the rate at which a grout can be injected into a crack is influenced by pressure and composition. Rate of flow is an index to the distance a grout can be pushed at a given pressure before friction causes the flow to cease. Under conditions dictating the use of low-pressure grouting, greater density of drilling than ordinary or the use of fluidifiers is indicated.

Relationship between pumping pressure, water-cement ratio, and crack thickness

37. An examination of fig. 4 shows that the crack thickness had a great effect on the lowest water-cement ratio grout that could be pumped through a crack. The pressure had little practical effect, since water-cement ratios lower than 0.5 are seldom employed.

Relationship between particle size of grout solids and width of crack penetrated

38. It will be noted that the lowest water-cement ratio neat grout

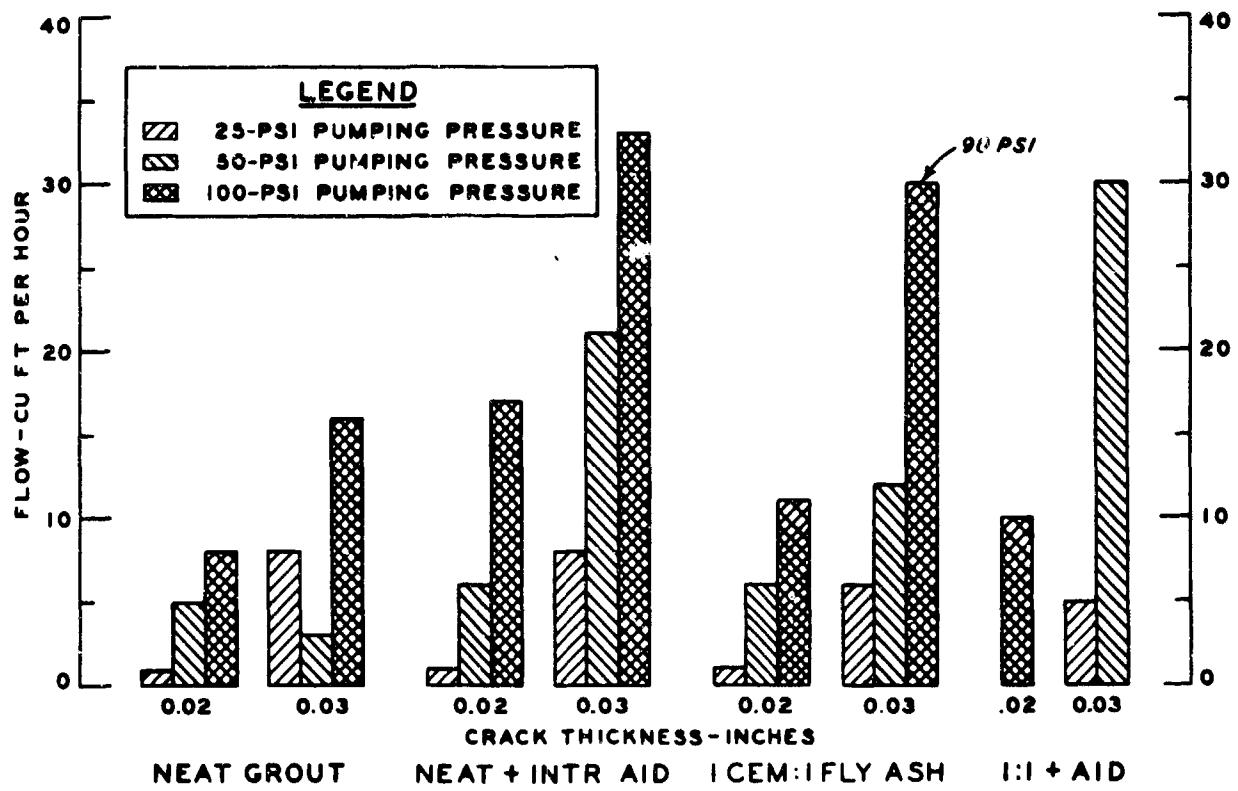


Fig. 15. Flow of 0.5 water-cement ratio grout at 25-, 50-, and 100-psi pumping pressures through 0.02- and 0.03-in. cracks

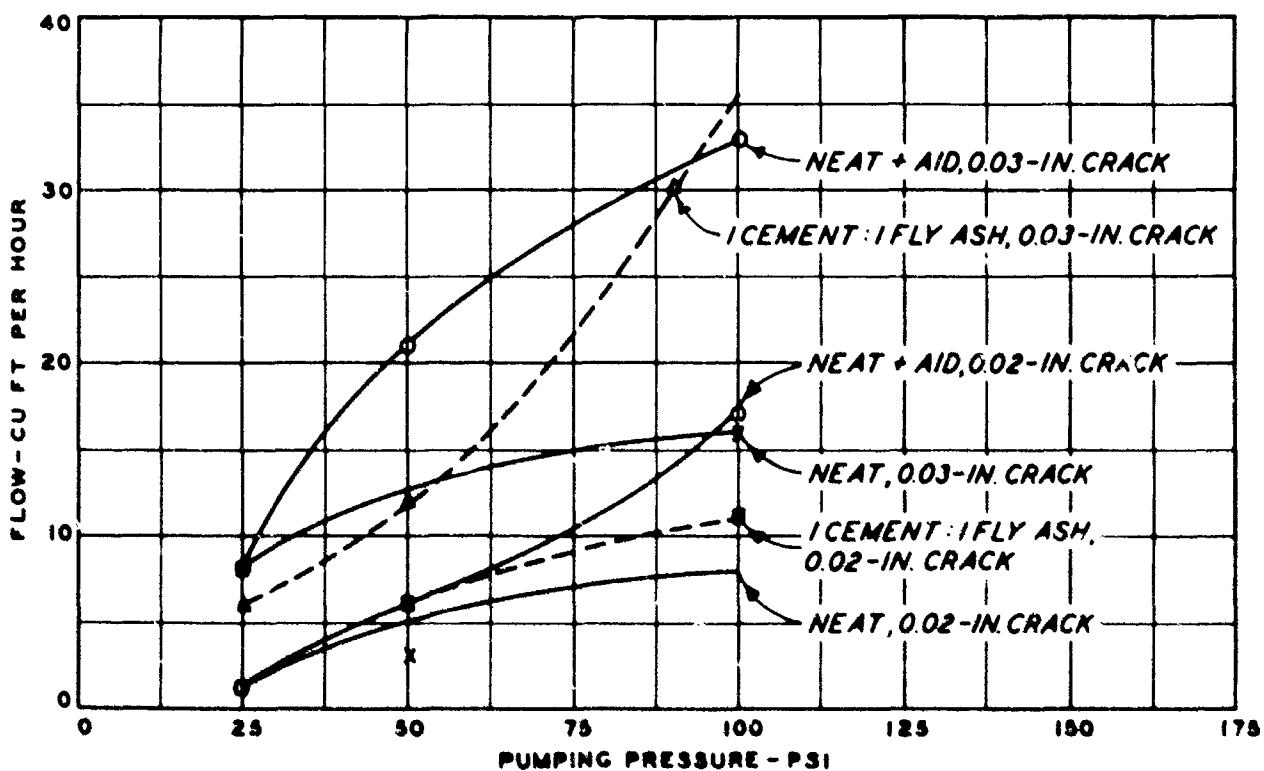


Fig. 16. Flow of 0.5 water-cement ratio grouts at the three pressures through 0.02- and 0.03-in. cracks

that could be forced through the 0.01 crack (fig. 4) was approximately 1.33 by weight. This is a dilute material of dubious quality and is believed to be neither durable nor impermeable. Grout containing fly ash could not be forced through the 0.01-in. crack even at considerably higher water-cement ratios. Examination of the physical data on the cement and fly ash shows that the maximum grain size of the cement was between 0.01 and 0.006 in., corresponding to the 50- and 100-mesh sieves with 0.1 per cent retained in this size range. The maximum grain size of the fly ash was somewhat in excess of 0.01 in. These results indicate that stoppage of the crack occurs when the crack-opening:grain-size ratio is 1.7 even with quite dilute grouts, and that with fly ash, where the maximum grain size exceeds the crack opening, grouting is impossible regardless of the water-cement ratio. The critical crack-opening:grain-size ratio probably varies with different materials, and it is believed that a safe value should exceed 3.0.

39. Alfred Machis* found in the grouting of sand that the ratio of pore diameter to grain size necessary to permit penetration of cement slurry was at least five. A. F. Taggart,** in his handbook, shows a graph that indicates the ratio of filter opening to maximum particle diameter for filter-cake formation. The fly ash had a maximum grain diameter somewhat larger than 250 microns. From the curve cited above, a filter-opening:grain-size ratio somewhat greater than three is indicated as necessary to prevent formation of a filter cake. The shape of the opening being grouted should influence the ratio of particle diameter to thickness. A slot-shaped opening, as used in the tests being reported, because of its relatively infinite width and no restricting sides, should permit a smaller ratio of particle-size diameter to opening to pass grout than a square or rounded opening of the same thickness.

* Alfred Machis, "Experimental observations on grouting sand and gravels," ASCE, Transactions, vol 113 (Nov 1948), pp 181-205.

** A. F. Taggart, Handbook of Mineral Dressing, Ores, and Industrial Minerals, New York, J. Wiley and Sons (1945).

Effect of Intrusion Aid
and RDA on penetration
characteristics of grouts

40. Fig. 4 shows that when Intrusion Aid was added to neat grout when grouting a 0.02-in. crack the resulting water-cement ratio was reduced 0.03 at 25-psi, 0.08 at 50-psi, and 0.17 at 100-psi pumping pressure, or an average reduction of 0.09 for all three pressures. When Intrusion Aid was used in 1 cement:1 fly ash grout, no reduction in water-cement ratio but rather an increase of 0.43 resulted in grouting the 0.02-in. crack at 25 psi. However, a 0.02 reduction in water-cement ratio resulted at 50 psi and a 0.09 reduction at 100 psi.

41. The use of Intrusion Aid in grouting the 0.03-in. crack with neat grout allowed a reduction in water-cement ratio of 0.07 at 25 psi, 0.11 at 50 psi, and an unknown amount at 100 psi since the cement plus Intrusion Aid grout at this pressure was not thickened to refusal. The use of Intrusion Aid in grouting the 0.03-in. crack with 1 cement:1 fly ash grout apparently caused an increased water-cement ratio of 0.03 at 25 psi, a decrease of 0.01 at 50 psi, and a decrease of 0.03 at 100 psi.

42. Fig. 4 also shows that the use of RDA in neat grout permitted a reduction of 0.01 at 25 psi, of 0.12 at 50 psi, and of 0.09 at 100 psi, or an average reduction of 0.07 in the minimum water-cement ratio that would penetrate a 0.02-in. crack. In grouting the 0.03-in. crack, the average reduction in water-cement ratio was 0.04 for the three pumping pressures used. The use of RDA in 1 cement:1 fly ash grout apparently allowed a reduction of 0.07 at 25 psi, of 0.03 at 50 psi, and an increase of 0.01 in the water-cement ratio at 100 psi when grouting the 0.02-in. crack. In grouting the 0.03-in. crack with cement-fly ash grout a slightly increased water-cement ratio was required for penetration at all three pressures used.

43. For penetration of a given crack thickness, Intrusion Aid seemed to be a little more efficient in lowering the water content of grout than RDA. That is, the Intrusion Aid was slightly more efficient than RDA as a fluidifier. However, little appears to be gained from use of either material except where it might be desirable to inject low

water-cement ratio grout under low pressure.

Quality of hardened grout
films from visual observation

44. Table 2 gives pertinent observations made when the two halves of the grouted specimen were opened and examined approximately 24 hr after pumping. The following tabulation summarizes these observations with the evaluations arranged in order of increasing water-cement ratios for each type of grout.

Type Grout	Water-cement Ratio, wt	Appearance	Bond Judged from Appearance
Neat	0.32-0.38	Good	Good to poor
	0.46	Good	Fair
	0.48	Fair	Poor
	0.51	Fair	Poor
	1.33	Poor, stringy	Poor
	1.34	Poor, stringy	Poor
Neat + Intrusion Aid	0.34	Good	Good
	0.34	Good	Fair
	0.35	Good	Good
	0.36	Good	Poor
	0.40	Good	Fair
	0.43	Good	Fair
	4.0	Poor, stringy	Poor
Neat + RDA	0.36	Good	Fair to poor
1:1 (cement:fly ash)	0.36	Good	Poor
	0.38	Good	Poor
	0.40	Good	Poor
	0.45	Fair, stringy	Poor
	0.49	Good	Poor
	4.0	Poor, stringy	Poor
	4.3	Poor, stringy	Poor
1:1 + Intrusion Aid (cement: fly ash + Aid)	0.33	Good	Poor
	0.37	Good	Poor
	0.40	Good	Poor
	0.43	Good	Fair
	0.43	Fair	Poor
	3.0	Poor, clumped	Poor
	4.3	Poor, stringy	Poor
1:1 + RDA (cement:fly ash + RDA)	0.035 to 0.038	Good	Good to poor
1.5:1 (cement:fly ash)	0.45	Fair, stringy	Poor

(Continued)

Type Grout	Water-cement Ratio, wt	Appearance	Bond Judged from Appearance
2:1 (cement:fly ash)	0.42	Fair, stringy	Fair
1.5:1 (cement:fly ash + RDA)	0.36	Good	Good
2:1 (cement:fly ash + RDA)	0.36	Good	Good
1:1 (cement:slag)	0.36	Good	Poor
1.5:1 (cement:slag)	0.38	Good	Poor
2:1 (cement:slag)	0.38	Good	Poor
1:1 (cement:pumicite)	0.56	Fair (soft)	Fair
1.5:1 (cement:pumicite)	0.50	Good	Fair
2:1 (cement:pumicite)	0.50	Good	Poor
1:1 (cement:opaline shale)	0.60	Fair (soft)	Poor
1.5:1 (cement:opaline shale)	0.55	Good	Poor
2:1 (cement:opaline shale)	0.55	Good	Poor

45. A grout film designated "good" in the tabulation was one which was hard to the fingernail, filled the cavity completely, and showed "none" to a "moderate" amount of bleeding as evidenced by channels cut by "bleed water" as it moved under slight residual pressure from the pumping process, or under gravity, toward points of lesser pressure or lower elevation. Fig. 17 shows a film designated "good." A film was designated "fair" when the grout could be scratched with the thumbnail or was traversed by numerous bleeding channels. It was called "poor"

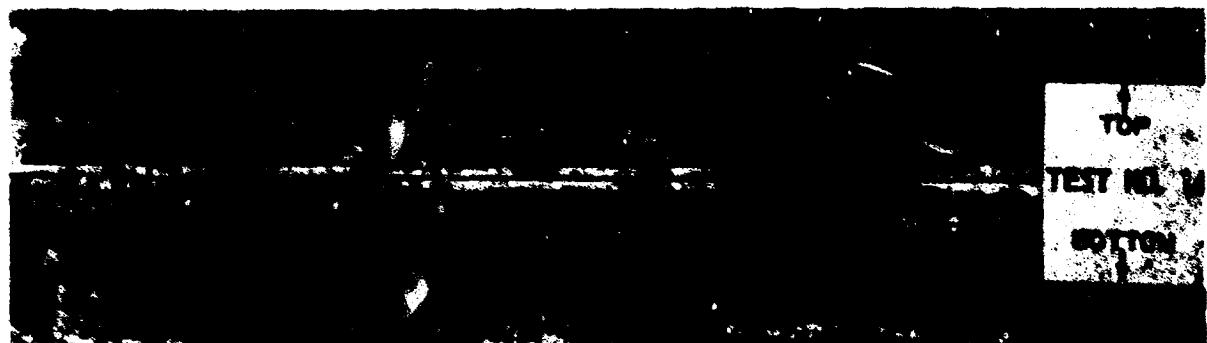


Fig. 17. Good quality, neat-cement-grout film from test 14, water-cement ratio 0.32, crack thickness 0.03 in., at a pumping pressure of 100 psi. Note white streaks caused by passage of water that was squeezed from the grout under the shim

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when it was quite soft or when the film occurred in lenses or streaks and could not be considered a continuous sheet of material. Fig. 18 shows a close-up of a poor-quality film.

46. Bond was considered "good" when approximately 40 per cent or more of the film adhered to the upper slab when the specimen was opened; "fair" when the adherence was approximately 25 to 40 per cent; and "poor" when the adherence was less than approximately 25 per cent.

47. It will be noted that the appearance of all the grouts having a water-cement ratio of 0.6 or less was "good" to "fair," and that where "poor" grout was encountered the ratio was 3.0 or higher. There are no intermediate values with the water-cement ratios ranging from 0.6 to 3.0, but it is only logical to assume that the quality of such films would have been "fair" to "poor." A direct comparison of the quality of different types of grout having the same water-cement ratio is not possible from the present data. However, judging from the bond evaluations, the use of Intrusion Aid appeared to be of some slight benefit in increasing adherence of the film to the top slab; but for grouting a horizontal opening between two nonabsorbent surfaces where a high-quality grout would be required, which would assuredly adhere to the top as well as the bottom of the cavity, a water-cement ratio not exceeding approximately 0.46 and neat grout should be used. The use of fly ash, judging from these qualitative data, may have had an adverse effect on the bond between top and bottom slab, although the quality of the films, from visual observation, was usually good. There was an apparent tendency toward clumping of the solids in some cases.

48. RDA in the cement-fly ash grouts tended to make them softer at 24 hr, but appeared to counteract to a large extent the tendency to agglomerate, and improved the bond to the top slab.

49. The use of slag, pumicite, and the opaline shale tended to improve the appearance of the grout films to a marked extent. Visual evidence of bleeding was entirely lacking with all the pumicite and opaline shale grouts and with the 1 slag:1 cement grout. The 1.5 cement:1 slag grout showed slight bleeding and the 2 cement:1 slag grout showed moderate bleeding. All three slag-grout films were hard



Fig. 18. Closeup (X2) of poor quality, neat-cement-grout film, test 18, water-cement ratio of 1.34, crack thickness 0.01 in., at a pumping pressure of 100 psi

to the fingernail at 24 hr; however, the 1 cement:1 pumicite grout could be dented by a fingernail at 24 hr and the 1 cement:1 shale grout could be slightly scratched by a fingernail at 24 hr. Bonding to the top slab did not seem to be improved by use of slag, pumicite, or opaline shale.

Strength of grout
films judged by shear tests

50. An attempt was made, during stage 2 of the tests, to measure the bond strength developed between top slab and the grout by making shear tests at 120 days age on sections of grouted specimens cut to 10-in. lengths. Three 0.01-in. cracks were grouted at 50 psi with various grouts as listed below, all with a water-cement ratio of 0.45 and with pressure maintained on the specimens for 15 min after pumping:

Type Grout	Test No.	Remarks
Neat + Intrusion Aid	46A	
Neat	48	
1 cement:1 fly ash	49	Came apart in handling
1 cement:1 fly ash	54	Came apart in handling
1 cement:1 fly ash + Aid	50	Came apart in handling

51. The following results were obtained:

Specimen Section	Shear Resistance, psi
46(1)	50
46(2)	Came apart in handling
46(3)	180
46(4)	70
48(1)	65
48(2)	30
48(3)	50
48(4)	60

Test values were quite low for the neat-grout and neat-grout plus Aid specimens tested, indicating very little bond. Those specimens containing fly ash did not develop enough bond to permit making a test.

PART IV: TESTS OF GROUT CHARACTERISTICS

Consistency

52. A satisfactory device for measurement of consistency is highly desirable and would enable the grouting crew to suit the quality and "pumpability" of the grout to the grouting conditions encountered. Consistency measurements were made on the grout itself by means of a piano-wire, pendulum-type viscosimeter, fig. 19, by a flow cone, and by a unit-weight device.

53. The viscosimeter is an instrument developed by Professor R. E. Davis of the University of California, Berkeley, and the Bureau of Reclamation. The viscosity or consistency of a slurry is measured by placing a sample in a shallow metal container mounted on a motor-driven turntable. A wire spider is suspended from a piano wire and is submerged in the sample of grout. A weight holds the piano wire taut. Torque is imparted to the spider and piano wire by the turning container of grout. The number of degrees of torque varies with the consistency of the sample being tested.

54. The flow cone is simply, as its name implies, a cone with a specified orifice from which a known volume of liquid flows in a certain length of time, depending upon its consistency. The flow cone used was one developed by the Prepekt Concrete Company and contained 1725 ml of grout. This amount of grout was discharged through a pipe orifice, 1-1/2 in. long by 1/2-in. inside diameter, in the bottom of the cone. Flow was timed

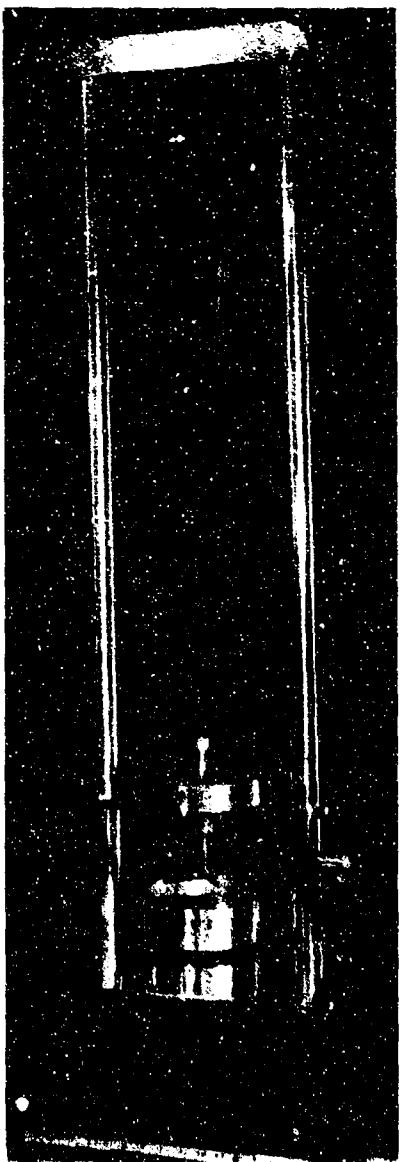


Fig. 19. Piano-wire viscosimeter

with a stop watch. In some of the tests the discharge pipe was modified to 6 in. in length and 3/8-in. inside diameter.

55. The unit weight device for consistency determinations was designed by the Ohio River Division Laboratories for use with neat-cement grout and consisted of a measure and specially graduated beam for translating unit weight into water-cement ratio. The device is described in Engineer Bulletin, Civil Works No. 48-4, 18 May 1948.

56. The piano-wire torque meter provided the most satisfactory means of measuring consistency in the laboratory. It was calibrated against the Stormer viscosimeter in connection with another investigation so that the readings obtained could be translated into standard

units of viscosity (poises). This calibration is shown on fig. 20. Consistency meter readings at fixed water-cement ratios for grout combinations used in the third-stage work are shown in table 3. For field work the torque meter might be too delicate since it would require a special indoor working area free of vibration.

57. For field work specially constructed flow cones with volume and discharge orifice integrated to produce sizable differences in readings with small changes in viscosity, or unit weight measures

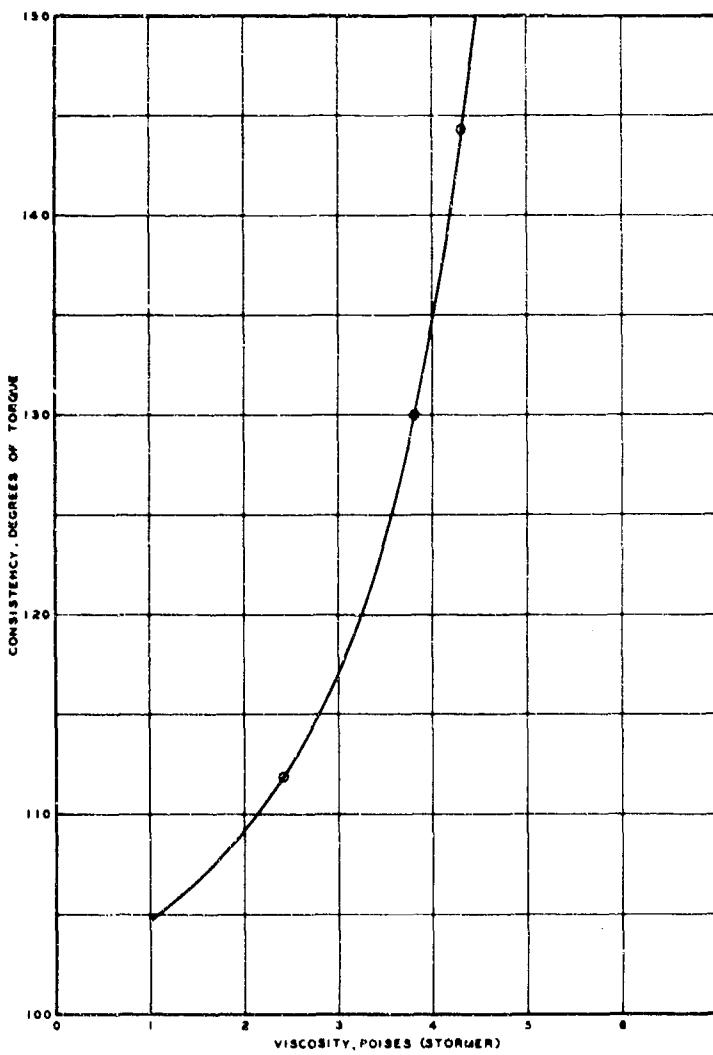


Fig. 20. Correlation between consistency measurements of torque meter and Stormer viscosimeter

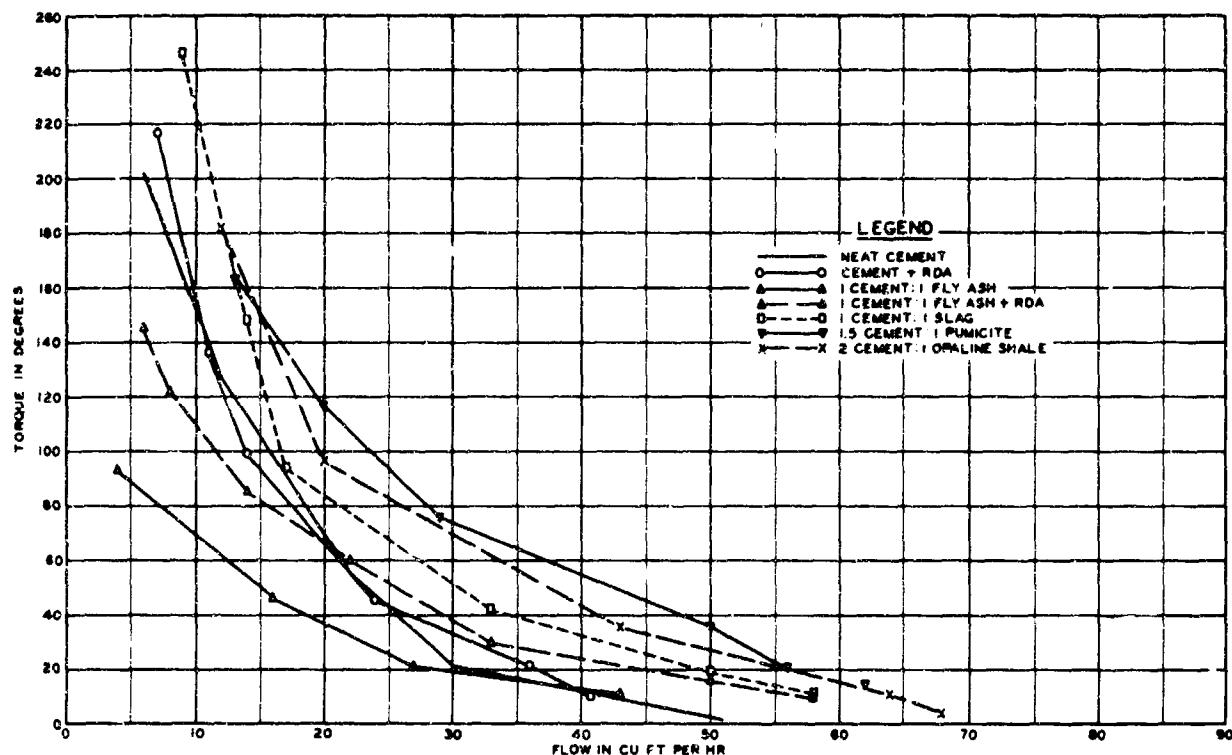


Fig. 21. Relationship between torque and flow of grouts through 0.03-in. crack at 50-psi pressure

with the unit weight of the grout expressed in grams per gallon would probably be satisfactory.

58. Whatever the method used for measuring viscosity, it should correlate with pumpability as measured in cubic feet per hour in order to have any practical meaning. Examination of fig. 21 shows good correlation between torque and pumpability (flow through the 0.03-in. crack at relatively constant pressure, approximately 50 psi) for several combinations of materials. There appeared to be little correlation between torque and flow when the grout combinations were considered collectively. This apparent lack of similarity of pumpability for grouts of equal viscosity through a thin crack is probably due to differences in grain-size distribution and particle shape of the solids in the grouts.

Bleeding

59. Two series of tests were carried out to determine the extent of bleeding of the grouts. The first series consisted simply of pouring

with a stop watch. In some of the tests the discharge pipe was modified to 6 in. in length and 3/8-in. inside diameter.

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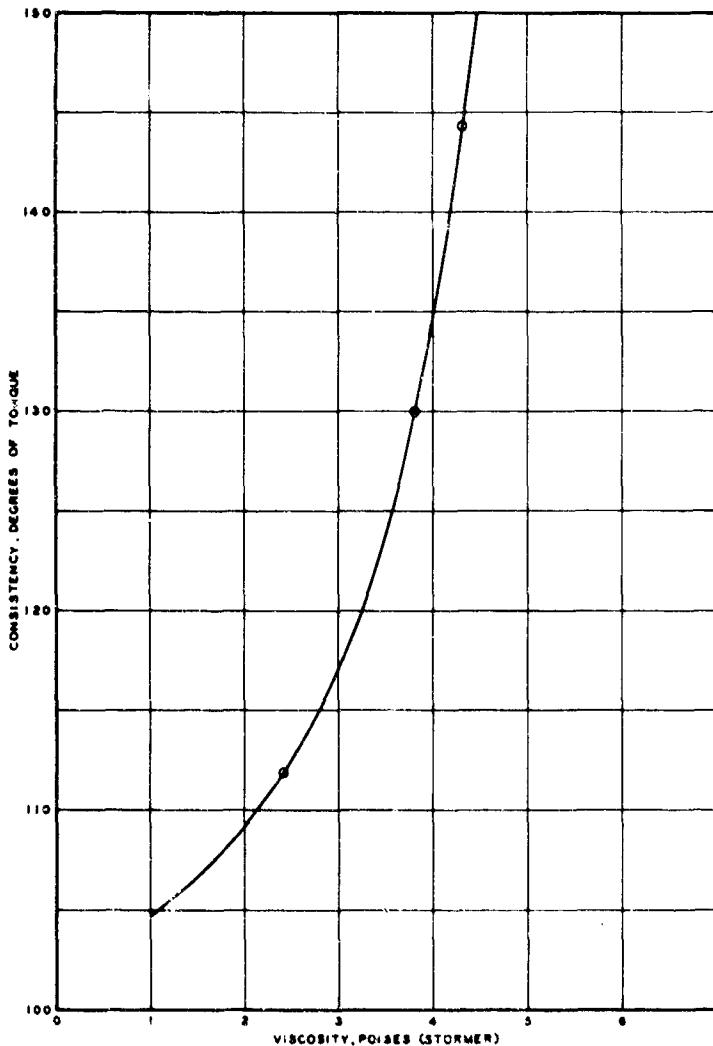


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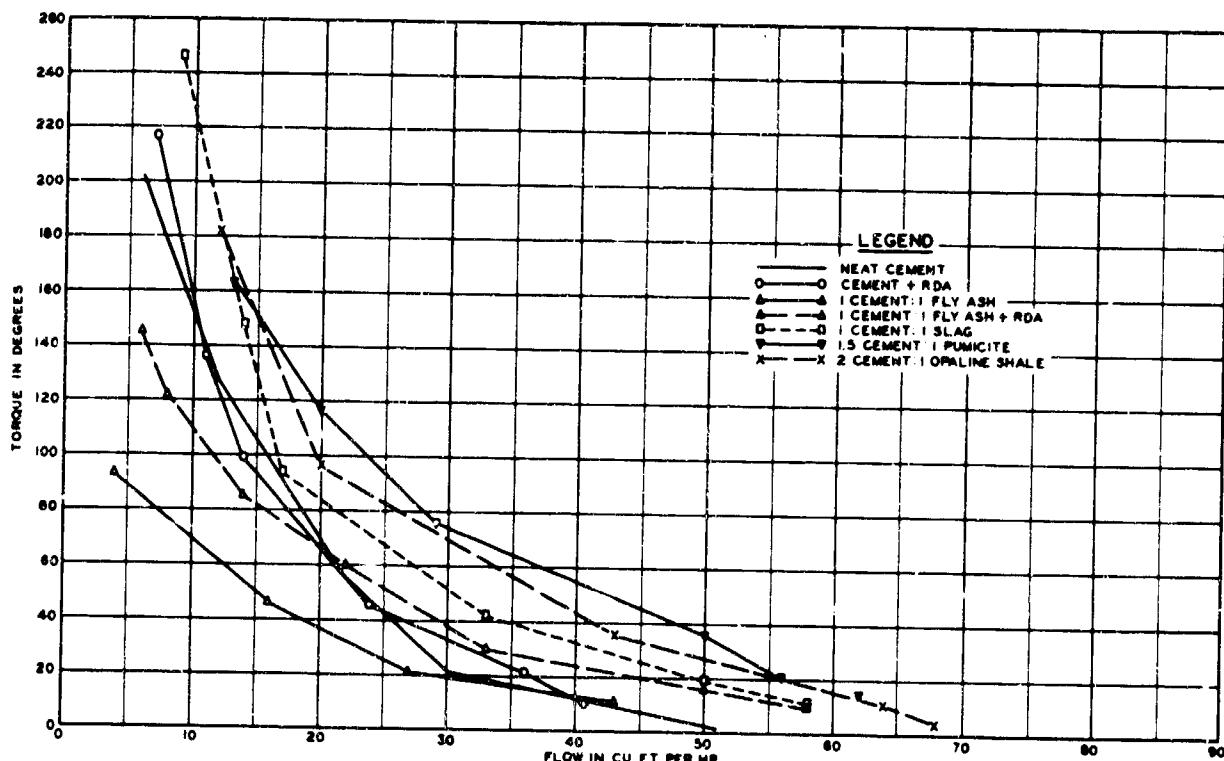


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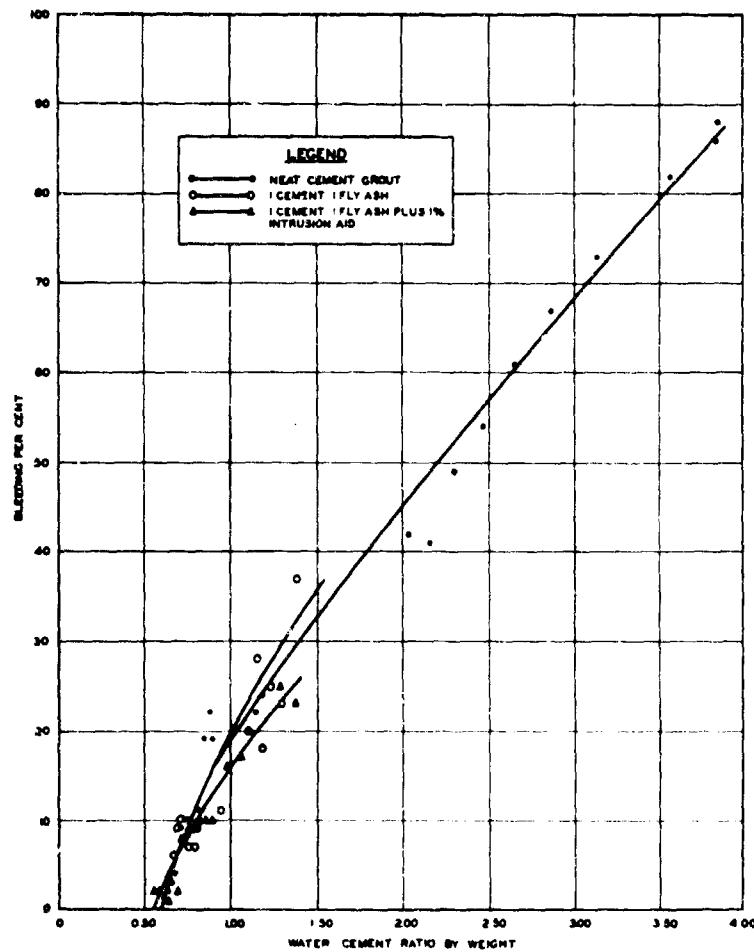


Fig. 22. Bleeding of grouts at one hour

500 ml of grout into a 1000-ml graduate and observing the separation of water with passage of time. The water-cement ratio was varied and measurements were made on neat grouts, 1 cement:1 fly ash grouts, and 1 cement:1 fly ash grouts with 1 per cent Intrusion Aid. Fig. 22 shows data obtained after one hour. Most of the bleeding had occurred at the end of one hour; no additional bleeding occurred after two hours. Use of Intrusion Aid in cement-fly ash grout appeared to slightly reduce its bleeding below that of neat and cement-fly ash grouts.

60. The second series of tests was performed according to ASTM C 243-52T insofar as practicable. The material combinations used were:

- a. Neat cement
- b. 1 cement:1 fly ash
- c. 2 cement:1 fly ash
- d. Cement + 0.23% RDA
- e. 1 cement:1 fly ash + 0.23% RDA
- f. 2 cement:1 fly ash + 0.23% RDA
- g. 1 cement:1 slag
- h. 2 cement:1 slag
- i. 1 cement:1 pumicite
- j. 2 cement:1 pumicite
- k. 1 cement:1 opaline shale
- l. 2 cement:1 opaline shale

61. A water-cement ratio of 0.4 was used in all cases except for the grouts containing opaline shale, which were too thick for use at 0.4, so 0.6 was used for these. Placing the grouts on approximately the same water-cement ratio basis regardless of solid material composition permitted evaluation of the effects of the constituents, other than water, on their bleeding characteristics. These data are shown in table 3.

62. RDA appeared to decrease sharply the rate and amount of bleeding

when used in neat grout. It appeared to act in a similar manner in the grouts with fly ash but to a lesser extent. Fly ash itself appeared to slow down the rate of bleeding but did not affect the total amount of bleeding. Slag appeared to lessen both the rate and amount of bleeding to a small extent. Both the pumicite and opaline shale sharply decreased the amount of bleeding and, to a somewhat lesser degree, the rate of bleeding.

Setting Time

63. Setting times of grouts containing the same constituents as used for the second series of bleeding tests, and made with both 0.4 and 0.8 water-cement ratios, were determined. The determinations were made by use of the 1-mm Vicat needle and small samples of grout placed in shallow wide-mouthed vials. The data obtained are shown in the tabulation below.

Grout	Water-cement Ratio of 0.4		Water-cement Ratio of 0.8	
	Setting Time, hr		Setting Time, hr	
	Initial	Final*	Initial	Final*
Neat cement	4 plus	Under 18	4 plus	Under 19
1 cement:1 fly ash	6 plus	Under 22	5 plus	Under 21
2 cement:1 fly ash	6 plus	Under 22	7 plus	Under 23
Cement + RDA	7 plus	Under 23	6 plus	72**
1 cement:1 fly ash + RDA	22**	Under 46	22**	30 to 46
2 cement:1 fly ash + RDA	22**	Under 46	26**	48**
1 cement:1 slag	6**	Under 22	8 plus	30**
2 cement:1 slag	4 plus	Under 20	5 plus	Under 21
1 cement:1 pumicite	4 plus	Under 20	6 plus	Under 22
2 cement:1 pumicite	4 plus	Under 20	5 plus	Under 21
1 cement:1 opaline shale	Too dry to test		6 plus	30**
2 cement:1 opaline shale	Too dry to test		5 plus	29**
Water-cement Ratio of 0.6†				
1 cement:1 opaline shale	3.5	Under 16*		
2 cement:1 opaline shale	6 plus	Under 21*		

* Exact setting time could not be obtained because of hours-of-work limitations.

** Approximate setting time.

† Fluid grouts could not be made at a 0.4 water-cement ratio using the opaline shale. The ratio of 0.6 was tested for information only; this ratio represents about the lowest ratio for fluid grout with this material.

64. Setting time is obviously important when grout is expected to cut off percolating water. In grouting by stages, it also controls the time that must elapse before holes can be cleared out and drilling resumed.

65. Setting times could not be determined exactly because of hours-of-work limitations. RDA appeared to lengthen the setting time by as much as three times that for the other grouts. The longest final setting time for any grout without RDA was approximately 30 hr (1 cement:1 opaline shale at 0.8 water-solids ratio). The longest setting time observed for the whole series was approximately 72 hr for neat grout with RDA at a 0.8 water-cement ratio.

Solubility

66. Two 50-ml portions of each of the selected grouts, listed in the 0.4 and 0.8 water:cement portions of the tabulation in paragraph 63,

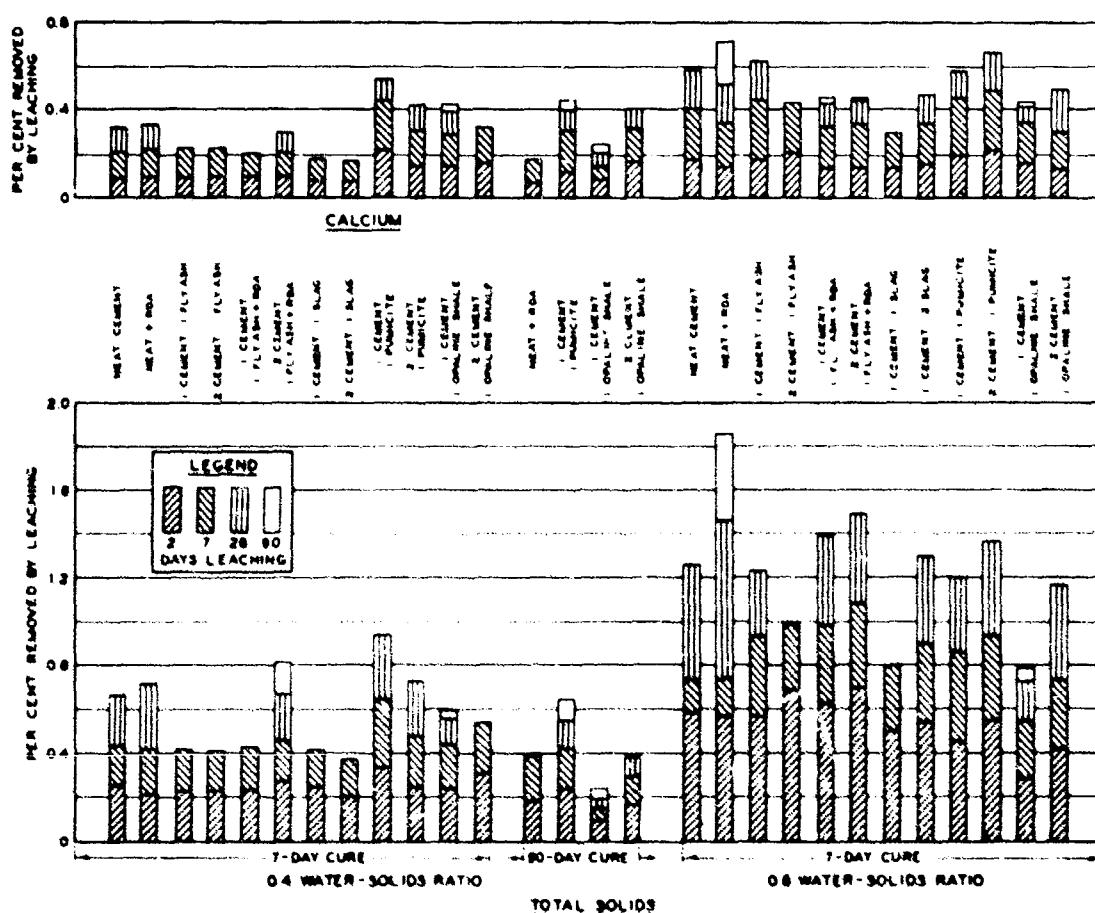


Fig. 23. Total solids and calcium leached from grouts

were placed in stoppered 200-ml flasks and one was cured for 7 days, the other for 90 days. At the end of the curing age, the samples were leached with 50 ml of distilled water for periods of 2, 7, 28, and 90 days. Leach water was then tested for pH, total solids, and calcium ion. The results of these tests are shown in table 4 with total solids and calcium expressed both in parts per million found in 200 ml of leach water and as a percentage based on the amount of solids and calcium in the original 50 ml of grout. The results are also shown graphically for total solids and calcium on fig. 23.

67. Unfortunately reaction occurred between the Pyrex glass flasks in which the grouts were cured and the alkalies in the grouts, causing many to crack after several weeks storage permitting the water to leak out. No test results are plotted on fig. 23 from specimens which were in the cracked flasks.

Conclusions

68. Conclusions drawn from the unspoiled tests are:

- a. Grouts exposed to leaching after only 7 days curing appeared to be somewhat more susceptible to leaching than those cured 90 days.
- b. About one-half of the dissolved material that leached out of the 0.4 water-solids ratio grouts was calcium, whereas about one-third of the dissolved material from the 0.8 water-solids ratio grouts was calcium.
- c. Increasing the water-solids ratio from 0.4 to 0.8 approximately doubled the amount of calcium and more than doubled the total solids in the leach water.
- d. RDA did not materially affect the solubility of the grouts made with a water-solids ratio of 0.4. Total solubility was increased and calcium solubility was decreased for the RDA grouts at a water-solids ratio of 0.8.
- e. Fly ash did not appear to materially affect solubility of the 0.4 ratio grouts. Solubility of the 0.8 ratio grouts seemed to be increased by use of fly ash.
- f. Behavior of RDA in the fly ash grouts was the same as in the neat-cement grout.
- g. Slag reduced the solubility of all constituents. The

effect on decreasing solubility was more marked at the higher water-solids ratio and was more pronounced for the 2 cement:1 slag than for the 1 cement:1 slag grout.

- h. Pumicite appreciably increased the over-all solubility of the 0.4 ratio grouts, but slightly decreased the solubility of the 0.8 ratio grouts. Calcium solubility was appreciably increased in the 0.4 ratio grouts and only slightly increased in the 0.8 ratio grouts.
- i. The opaline shale materially reduced the solubility of all constituents. Total solids were reduced by about 50 per cent and calcium by about 40 per cent.

PART V: SUMMARY OF RESULTS

69. The reader should keep in mind in evaluating the data that the investigation described comprises only a single set of tests per condition.

Factors Influencing Penetration of GroutsSurface texture

70. It was found that the surface condition of the specimens, that is, whether the surfaces to be grouted were smooth or roughened, had a pronounced effect on how thick a grout could be forced through the cracks. The smoother surfaces permitted the use of considerably lower water-cement ratios than did the roughened surfaces for all three crack thicknesses tested.

Water-cement ratio

71. The water-cement ratio influenced the thickness of crack that could be penetrated at a given pumping pressure. Neat grout with a water-cement ratio of 0.43 would penetrate the 0.03-in. crack at 25 psi, but the water-cement ratio had to be increased to 2.67 before neat grout would penetrate the 0.01-in. crack at the same pressure.

Pumping pressure

72. An approximately straight-line pressure drop occurred along the specimens while the 0.02- and 0.03-in. cracks were being grouted. The pressure was either 25, 50, or 100 psi where the grout entered, and was 0 psi where it emerged with intermediate pressures measured along the length of the specimen. The pressure drop was steeper for the first 12 in. of specimen length than for the remaining 36 in. when the 0.01-in. crack was being grouted. The steepness of the gradient for the first 12 in. increased as the grout thickened and the flow decreased.

73. Increased pumping pressures caused directly proportionate increases in grout flow. At identical water-cement ratios, pressures, and crack thicknesses the neat-cement grout had least flow, cement-fly ash grout had more flow, and neat grout plus Intrusion Aid had still more flow. However, the flow was not greatest, as might be expected, with

cement-fly ash grout plus Intrusion Aid. Data on the cement-fly ash-Intrusion Aid grout were anomalous and inconclusive.

74. When the crack width was 0.02 or 0.03 in. the minimum water-cement ratio (thickness of grout) the crack would accept was little affected by the grouting pressure, however the thickness of the grout that would penetrate the 0.01-in. crack depended somewhat on pressure. At 25 psi, the thickest grout that would penetrate the 0.01-in. crack had a water-cement ratio of 2.67. When the pressure was increased to 50 psi, grout with a water-cement ratio of 1.33 would penetrate, but a further increase in pumping pressure to 100 psi did not permit use of still thicker grout.

Grain size

75. The use of a No. 50 sieve cloth for straining the grout seemed beneficial and probably the use of a No. 60, or even a No. 100 cloth, if vibrated, would be practical in removing oversized particles which cause trouble in grouting the tightest seams. The fly ash contained grains of material larger than 0.01 in. and grouts containing fly ash would not penetrate the 0.01-in. fissures. Based on the maximum grain size of the cement, 0.006-0.01 in., and the thinnest cracks grouted, 0.01 in., the ratio of crack thickness to grain size should not be less than 1.7, and probably safer ratios would be 3.0 or more (see paragraphs 38 and 39).

76. The use of fly ash appears limited to grouting larger cracks unless a supply of fine material can be obtained, or unless fly ash can be processed, possibly by air separation, to remove the larger granules. No attempt was made to grout 0.01-in. cracks with grout containing ground slag, pumicite, or opaline shale. Difficulty would doubtless have been encountered with the slag and shale whose grain size exceeded 0.01 in.

77. The use of more finely ground cements, such as high-early strength cement, or ordinary cement processed through an air separator might be feasible.

Chemical fluidifiers

78. The use of Intrusion Aid appeared to be a help in reducing the water ratio of the grout that could be pumped through cracks of 0.03-in. thickness with roughened surface, and 0.03- and 0.02-in. thickness with

smooth surfaces. It is believed that the Aid should also have promoted use of lower water-cement ratio grout for the roughened 0.02-in. crack but did not in these tests because of surface factors in the specimens. Intrusion Aid was of no help in promoting penetration of 0.01-in. cracks at any water ratio tried because of the limits of penetration placed upon the grout containing fly ash by the coarseness of the fly ash. Penetration of 0.01-in. cracks with neat grout containing Intrusion Aid was not tried. The use of Intrusion Aid appeared to cause a small reduction in bleeding and a slight expansion in the grouts.

79. Intrusion Aid and RDA appeared to increase the fluidity of the neat grouts so that a seam of given thickness would pass a greater amount of such grout than of plain grout having the same water-cement ratio; conversely, grout with fluidifiers, at a lower water-cement ratio and at a given pressure, would penetrate a given crack better than grouts without fluidifiers.

80. Intrusion Aid seemed to be more effective with neat grouts than with grouts containing fly ash. The cement-fly ash grout with Intrusion Aid did not penetrate either the 0.02- or the 0.03-in. seams significantly better than the same grouts without Aid.

Mineral fines

81. Fly ash appeared to increase the fluidity of the grouts in which it was used. The ground slag also appeared to increase fluidity, however this was not conclusive. The pumicite and opaline shale both greatly stiffened the grouts in which they were used; however, at comparable consistency with neat grout, the pumicite and opaline shale grouts pumped better than the neat grout. At equal consistency the fly ash and slag grouts pumped about the same as neat grout.

Factors Affecting Quality of Grout Films

Water-cement ratio

82. The grout films having water-cement ratios of 0.5 or less appeared hard and filled the fissures completely with little evident bleeding. However, the bond of top to bottom half of the grouted specimen

was not considered good except when water-cement ratios were lower than 0.4.

83. Bleeding occurs in grouts having water ratios in excess of 0.50, and becomes more severe the higher the unit water content. A narrow band of dense hard grout was always found next to the shim beneath which water had escaped. This band of dense grout was always narrow, never exceeding 3/4 in. in width, no matter what the original water content. It has frequently been hypothesized that the original water content of a grout is not very important as the excess water is squeezed out into the pores of the rock and extremely fine seams (too fine to accept grout) leaving a dense hard material filling the cavity. It was not found possible to squeeze this water out with the 100-psi grouting pressure used, in the 10 to 15 minutes during which pressure was maintained, except in a narrow band around the edge of the specimen, even with the outer edges of the specimens open to the air at 0 (relative) pressure.

84. Setting time increased with water content. Neat grout with a water content as high as 3.8 required ten days to set in the setting-time tests, and had not set in 13 days when spread into a thin film in the grouting tests. Such thin grout containing Intrusion Aid did not set in 14 days in the setting-time tests. These thin grouts can be shown to be incapable theoretically of developing any compressive strength; see appendix B. The higher water-solids ratio grouts were also much less resistant to leaching than those with lower ratios.

Chemical fluidifiers

85. Bonding of the hardened grout films to the top slab was poor (25 per cent or less of film adhering to top) for all grouts except those containing fly ash with RDA where the bond was considered good (40 per cent or more adhering to top). Bond strength as measured by shear tests on neat and neat-cement plus Intrusion Aid grouts was relatively low although the use of Intrusion Aid appeared to somewhat increase bonding between top and bottom halves of the specimen in neat grout. RDA did not appear to benefit the bond condition of specimens intruded with neat grout.

86. Both Intrusion Aid and RDA prolonged setting time and reduced bleeding. The solids in the fly ash grouts tended to clump or agglomerate. This tendency appeared to be reduced but not eliminated by the fluidifier. Intrusion Aid was not used in the tests for solubility of grout films, but RDA had little effect on the leaching characteristics of the grouts tested.

Mineral fines

87. Hardened grout films containing fly ash showed evidence of channeling action and apparent agglomeration. This tendency was not noted when the slag, pumicite, and opaline shale were used. The use of ground slag, pumicite, and opaline shale greatly improved the apparent quality of the grout film, virtually eliminating evidence of bleeding, channeling, and agglomeration. Bond between top and bottom halves of the specimens was not improved by use of the several mineral fines.

88. The use of fly ash had little apparent effect on the solubility of the 0.4 water-ratio grouts, but appeared to increase the solubility of the 0.8 water-ratio grout. Slag reduced the leaching in all cases with the most pronounced effect in the higher water-ratio grouts. Pumicite increased the over-all solubility of the 0.4 ratio grout but slightly decreased the over-all solubility of the 0.8 ratio grout. The opaline shale greatly reduced the over-all solubility of both the low- and high-water-solids ratio grouts in which they were used.

Curing

89. Grout films cured for 90 days appeared more resistant to leaching than those cured 7 days.

Measurement of Consistency

90. The measurement of consistency was readily accomplished in the laboratory with the torsion viscosimeter. Changes in consistency can also be detected and controlled readily by means of unit weight measurements. The flow cone was not too sensitive to small changes in viscosity although a cone with an orifice restricted enough to require more time to discharge might prove suitable for field use. The torsion

viscosimeter, because it is delicate and requires a quiet, level area for operation, would not be as suitable for field as for laboratory use.

PART VI: CONCLUSIONS

91. The surface texture of a fissure has a distinct influence on the thickness of grout that can be used to fill it. The smoother the surface the lower the water-cement ratio of the grout can be and still penetrate the crack.

92. The maximum grain size of the solids in the grout determines the minimum crack width that can be grouted. The ratio of crack width to grain size should probably be three or more.

93. The use of such materials as Intrusion Aid and RDA increases the fluidity of grouts, to some extent, thereby promoting penetration of a given crack width with grouts of a slightly lower water-cement ratio than could be used successfully without them. Bleeding can be reduced somewhat and setting time increased by use of these materials.

94. It is impracticable to squeeze the excess water from a thin grout, thereby leaving a dense, hard filler in the cavity, at the pressures used in this program.

95. Bleeding largely prevents bonding of the grouting material to the upper surface of the fissure.

96. The use of finely ground mineral admixtures such as granulated blast-furnace slag, pumicite, and opaline shale can reduce the bleeding of a grout and greatly improve the continuity and appearance of the hardened grout film.

97. The solubility of a grout film is influenced by the water-cement ratio and composition of the film and length of curing. Certain mineral admixtures can be used to reduce the amount of leaching that a grout film undergoes.

98. A straight-line pressure gradient occurs along a fissure being grouted only if the crack is of sufficient width.

Table 1
Results of Grout Pumping Tests

Serial No.	Test No.	Stage No.	Grout	Crack thickness in.	Water-cement ratio (by Wt)	Flow cu ft/hr	Pressure (psi) Gage No.				Consistency (Torque Deg)	Remarks
							0(A)	1	2	3		
1	1	1	Neat cement	0.02	0.45	----	100	--	--	--	0	---
2	9	1	Neat cement	0.01	3.73	8	100	--	--	--	0	---
					3.34	7	100	--	--	--	0	---
					3.03	5	100	--	--	--	0	---
					2.76	4	100	--	--	--	0	---
					2.55	3	100	--	--	--	0	---
					2.36	2	100	--	--	--	0	---
					2.20	1.5	100	--	--	--	0	---
					2.06	0.9	100	--	--	--	0	---
					1.93	0	100	--	--	--	0	---
3	11	1	Neat cement	0.01	7.6	2	100	--	--	--	0	---
					Refused grout. Preliminary test. Roughened surfaces on specimen							
4	5	1	Neat cement	0.02	1.08	14	100	--	--	--	0	---
					1.04	7	100	--	--	--	0	---
					1.02	0	100	--	--	--	0	---
5	2	1	Neat cement	0.03	----	--	--	--	--	--	0	Test voided
6	4	1	Neat cement	0.03	0.58	34	100	--	--	--	0	Repeat of test 2. Roughened surfaces on specimen
					0.55	30	100	--	--	--	0	---
					0.53	26	100	--	--	--	0	---
					0.52	22	100	--	--	--	0	28*
					0.50	6	100	--	--	--	0	36
7	13	1	Neat cement	0.03	0.70	69	80	--	--	--	0	---
					0.62	64	80	--	--	--	0	---
					0.57	62	80	--	--	--	0	---
					0.52	49	38	--	--	--	0	---
					0.46	25	80	--	--	--	0	---
					0.42	12	80	--	--	--	0	---
					0.40	5	30	--	--	--	0	---
					0.39	4	100	--	--	--	0	---
8	18	1	Neat cement	0.01	2.05	9	100	--	--	--	0	---
					1.96	6	100	--	--	--	0	---
					1.76	4	100	--	--	--	0	---
					1.60	3	100	--	--	--	0	---
					1.34	0	100	--	--	--	0	---
9	17	1	Neat cement	0.02	1.01	31	100	--	--	--	0	---
					0.77	25	100	--	--	--	0	---
					0.73	20	100	--	--	--	0	---
					0.64	15	100	--	--	--	0	---
					0.57	11	100	--	--	--	0	---
					0.54	6	400	--	--	--	0	---
					0.52	0	100	--	--	--	0	---
10	14	1	Neat cement	0.03	0.51	48	80	--	--	--	0	---
					0.45	37	80	--	--	--	0	---
					0.41	27	80	--	--	--	0	---
					0.38	25	78	--	--	--	0	---
					0.35	12	80	--	--	--	0	---
					0.32	5	80	--	--	--	0	---
					0.30	0	80	--	--	--	0	---
11	46	2	Neat cement	0.03	0.50	41	100	62	40	32	0	---
					0.50	40	100	56	36	22	0	Pump 80 strokes per min
					0.45	35	100	66	46	25	0	Pump 68 strokes per min
					0.42	27	100	72	50	28	0	Pump 68 strokes per min
					0.38	19	100	75	47	26	0	Pump 68 strokes per min
												Pump 72 strokes per min
12	34	2	Neat cement	0.01	4.00	6	50	30	19	9	0	---
					3.00	5	50	30	17	7	1	---
					2.00	4	50	21	15	5	0	---
					1.50	3	50	21	12	5	0	---
					1.33	0	50	9	6	4	0	---
13	35	2	Neat cement	0.02	1.33	25	50	31	21	11	0	---
					1.10	21	50	32	22	12	0	---
					0.92	18	50	32	22	12	0	---
					0.71	11	50	34	24	12	0	---
					0.57	6	50	27	25	13	0	---
					0.48	0	50	--	--	--	0	---
14	35	2	Neat cement	0.03	0.48	0	50	25	16	8	0	Refuse grout at initial water-cement ratio
15	37	2	Neat cement	0.03	1.20	55	50	35	25	15	0	---
					0.86	44	50	28	20	12	0	---
					0.67	26	50	23	17	9	0	---
					0.57	5	50	7	5	1	0	---
					0.52	4	50	8	6	2	0	---
					0.46	2	50	8	6	2	0	---
16	43	2	Neat cement	0.03	0.45	--	50	--	--	--	0	Grout not thickened to refusal, shear test specimen, static pressure maintained 15 min after pumping
17	52	2	Neat cement	0.03	2.0	--	40	40	40	40	40	Grout not thickened to refusal, static pressure applied for approx 1 hr to attempt to squeeze water from grout

(Continued)

* Consistency measured on torque meter (piano-wire viscosimeter) with spider wire of 0.125-in. diameter.

Table 1 (Continued)

Serial No.	Test No.	Stage No.	Grout	Crack thickness in.	Water-cement Ratio (by Wt)	Flow cu ft/hr	Pressure (psi) Gage No.				Consistency (Torque Deg)	Remarks
							0(A)	1	2	3		
18	75	3	Neat cement	0.03	1.00	53	40	28	22	14	0	11* Valve 4 closed and pressure maintained for 10 min after pumping
					0.71	30	50	18	15	8	0	
					0.55	22	50	23	18	9	0	
					0.45	12	50	27	21	10	0	
					0.42	8	50	29	21	10	0	
					0.38	5	50	28	18	9	0	
19	25	2	Neat cement	0.01	4.00	4	24	12	9	3	0	---
					3.44	2	28	12	9	2	0	
					2.67	0.3	25	0	0	0	0	
					1.71	15	25	18	13	7	0	
20	26	2	Neat cement	0.02	1.34	14	25	13	12	7	0	---
					0.92	10	25	20	13	7	0	
					0.71	6	25	19	13	7	0	
					0.57	2	25	19	13	7	0	
					0.48	1	25	23	17	10	1	
					0.46	0	25	20	14	10	2	
21	27	2	Neat cement	0.03	1.00	32	25	18	13	8	0	---
					0.75	24	25	18	13	7	0	
					0.60	16	25	17	13	7	0	
					0.50	8	25	17	13	7	0	
					0.46	6	25	19	13	6	0	
					0.43	4	25	19	12	6	0	
22	10	1	1 cement:1 fly ash	0.01	7.6	--	100	--	--	--	0	Roughened surfaces on specimen. Refused grout
					4.3	--	--	--	--	--	0	
23	21	1	1 cement:1 fly ash	0.01	4.3	--	--	--	--	--	0	Refused grout
					4.3	--	100	--	--	--	0	
25	7	1	1 cement:1 fly ash	0.02	1.13	9	100	--	--	--	0	Roughened surfaces on specimen
					1.12	5	100	--	--	--	0	
					1.05	2	100	--	--	--	0	
					1.00	0	100	--	--	--	0	
26	20	1	1 cement:1 fly ash	0.02	0.95	21	100	--	--	--	0	---
					0.79	18	100	--	--	--	0	
					0.70	15	100	--	--	--	0	
					0.65	13	100	--	--	--	0	
					0.59	11	100	--	--	--	0	
					0.52	6	100	--	--	--	0	
27	20-A	1	1 cement:1 fly ash	0.02	0.95	21	100	--	--	--	0	---
					0.85	32	100	--	--	--	0	
					0.66	20	100	--	--	--	0	
					0.53	10	100	--	--	--	0	
					0.48	7	100	--	--	--	0	
					0.46	5	100	--	--	--	0	
28	3	1	1 cement:1 fly ash	0.03	0.64	5	50	--	--	--	0	Roughened surfaces on specimen. static pressure on specimen 15 min
					0.64	7	78	--	--	--	0	
					0.64	10	94	--	--	--	0	
					0.64	20	45	--	--	--	0	
					0.63	2	50	--	--	--	0	
					0.60	5	60	--	--	--	0	
29	15	1	1 cement:1 fly ash	0.03	0.64	78	75	--	--	--	0	---
					0.59	72	76	--	--	--	0	
					0.52	62	72	--	--	--	0	
					0.47	18	40	--	--	--	0	
					0.44	16	50	--	--	--	0	
					0.42	13	50	--	--	--	0	
30	16	1	1 cement:1 fly ash	0.03	0.40	10	54	--	--	--	0	Duplication of test 15
					0.38	5	75	--	--	--	0	
					0.36	0	95	--	--	--	0	
					0.6	4	75	--	--	--	0	
					0.50	44	75	--	--	--	0	
					0.52	32	80	--	--	--	0	
31	45	2	1 cement:1 fly ash	0.02	0.47	26	80	--	--	--	0	---
					0.44	22	80	--	--	--	0	
					0.42	19	90	--	--	--	0	
					0.40	17	90	--	--	--	0	
					0.38	14	95	--	--	--	0	
					0.37	5	108	--	--	--	0	
					0.36	0	100	--	--	--	0	---

(Continued)

* Consistency measured on torque meter with spider wire diameter increased to 0.5625 in.

Table 1 (Continued)

Serial No.	Test No.	Stage No.	Grout	Crack thickness in.	Water-cement ratio (by Wt)	Flow cu ft/hr	Pressure (psi) Gage No.					Consistency (Torque Duz)	Remarks
							0(A)	1	2	3	4(B)		
32	39	2	1 cement:1 fly ash	0.03	0.60 0.50 0.46 0.43 0.40 0.36	26 15 11 8 5 2	50 50 50 50 50 50	37 36 35 35 35 36	24 24 23 23 23 24	14 14 14 14 14 14	0 0 0 0 0 0	---	0.36 water-cement ratio grout was as thick as pump could handle
33	49	2	1 cement:1 fly ash	0.03	0.45	--	50	--	--	--	--	---	Shear test specimen came apart in handling
34	54	2	1 cement:1 fly ash	0.03	0.45	--	50	--	--	--	--	---	Repeat of test 49. Shear test specimen. Static pressure 15 min
35	76	3	1 cement:1 fly ash	0.03	1.00 0.71 0.55 0.45	43 27 16 4	50 50 50 50	26 26 21 24	18 14 12 13	10 7 7 5	0 0 0 0	11 ^a 21 46 93	
36	72	2	1 cement:1 fly ash	0.03	1.00 0.75 0.67 0.60 0.56 0.50 0.45 0.43 0.40	41 31 26 20 16 11 8 5 2	50 50 50 50 50 50 50 50 50	31 32 34 36 37 37 38 36 36	21 21 23 24 25 25 25 25 27	12 11 12 11 12 12 14 13 14	0 0 0 0 0 0 0 0 0	---	
37	28	2	1 cement:1 fly ash	0.01	4.0	0	25	0	0	0	0	---	Refused grout
38	29	2	1 cement:1 fly ash	0.02	1.50 0.92 0.80 0.71 0.57 0.52 0.52	12 6 4 3 2 1 0	25 25 25 25 25 25 25	17 15 14 13 13 13 24	12 10 10 10 17 19 19	7 6 5 5 10 11 21	0 0 0 0 0 0 19	---	Pressure maintained on specimen 10 min after refusal
39	30	2	1 cement:1 fly ash	0.03	1.50 1.00 0.75 0.60 0.50 0.46 0.43 0.40 0.40 0.40	39 30 19 12 6 5 3 2 0	25 25 25 25 25 25 25 25 25	19 18 18 17 16 17 18 18 18	13 13 12 12 12 12 13 13 13	8 7 6 6 6 7 7 7 7	0 0 0 0 0 0 0 0 0	---	Static pressure applied approx 10 min after refused at 25 psi. Pressure increased and flow started. Static pressure applied for approx 10 min after resumption of flow at increased pressure
40	64	2	1 cement:1 fly ash	0.03	0.45	--	5	--	--	--	--	---	Half of specimen assembly was 3/4-in.-thick plexiglas to permit observation of grout flow pattern
41	77	3	1.5 cement:1 fly ash	0.03	1.00 0.71 0.55 0.45	51 37 15 6	50 50 50 50	26 20 12 12	19 14 9 9	10 7 3 2	0 0 0 0	9 ^a 21 45 91	
42	78	3	2 cement:1 fly ash	0.03	1.00 0.71 0.55 0.45 0.42	55 49 33 17 12	50 50 50 50 50	36 37 35 32 32	30 31 15 31 32	17 16 15 12 12	0 0 0 0 0	12 ^a 20 45 95 125	
43	83	3	1 cement:1 slag	0.03	1.00 0.71 0.55 0.45 0.42 0.38 0.36	50 50 33 17 14 9 4	50 50 50 50 50 50 50	32 33 33 33 33 35 35	20 26 26 28 28 26 24	15 14 12 12 12 12 12	0 0 0 0 0 0 0	11 ^a 19 42 94 148 246 431	
44	84	3	1.5 cement:1 slag	0.03	1.00 0.71 0.55 0.45 0.42 0.38	62 51 33 11 6 1	50 50 50 50 50 50	33 47 27 21 18 18	27 29 29 33 33 33	11 9 6 7 6 6	0 0 0 0 0 0	20 ^a 25 51 106 167 219	
45	85	3	2 cement:1 slag	0.03	1.00 0.71 0.55 0.45 0.42 0.38	51 40 26 15 10 7	50 50 50 50 50 50	33 30 32 32 32 34	21 19 20 19 20 20	30 28 26 26 26 26	0 0 0 0 0 0	18 ^a 26 46 100 153 234	
46	86	3	1 cement:1 pumicite	0.03	1.00 0.71 0.55	62 41 18	50 50 50	34 33 32	38 36 36	13 11 10	0 0 0	---	

(Continued)

* Consistency measured on torque meter with spider wire diameter increased to 0.9625 in.

Table 1 (Continued)

Serial No.	Test No.	Stage No.	Grout	Crack thickness in.	Water-cement ratio (by wt)	Flow cu ft/hr	Pressure (psi) Gage No.					Consistency (Torque Deg)	Remarks
							0(A)	1	2	3	4(B)		
47	87	3	1.5 cement:1 pumicite	0.03	2.00 1.20 0.86 0.66 0.60 0.55 0.50	62 60 50 29 20 13 7	50 50 50 50 50 50 50	32 30 32 32 32 32 24	22 18 20 18 12 10 12	13 12 12 10 8 10 10	0 0 0 0 0 0 0	15* 20 36 76 117 161 316	
48	88	3	2 cement:1 pumicite	0.03	2.00 1.20 0.86 0.66 0.60 0.55 0.50	50 43 32 21 15 9 7	50 50 50 50 50 50 50	30 23 23 26 28 29 32	18 12 13 4 4 4 3	10 8 8 8 9 9 9	0 0 0 0 0 0 0	---	
49	89	3	1 cement:1 opaline shale	0.03	2.00 1.20 0.86 0.66 0.60	64 61 32 9 3	50 50 50 50 50	36 32 35 32 26	24 22 22 18 17	14 12 11 11 11	0 0 0 0 0	---	
50	90	3	1.5 cement:1 opaline shale	0.03	2.00 1.20 0.86 0.66 0.60 0.55	60 56 35 15 10 4	50 50 50 50 50 50	34 32 31 32 32 31	20 20 20 22 22 <br;>22</br;>	14 13 11 11 11 12	0 0 0 0 0 0	---	
51	91	3	2 cement:1 opaline shale	0.03	2.00 1.20 0.86 0.66 0.60 0.55	68 64 43 20 12 6	50 50 50 50 50 50	35 32 33 32 31 31	20 20 20 20 20 20	12 11 11 10 10 10	0 0 0 0 0 0	4* 11 36 96 182 ---	
52	46	2	1 cement + 1% Intrusion Aid	0.02	1.50 0.64 0.50 0.43 0.39 0.37 0.34	40 27 17 9 7 6 3	100 100 100 100 100 100 100	66 70 70 67 65 65 62	42 48 48 45 30 30 27	25 25 25 25 25 18 16	0 0 0 0 0 2 0	---	
53	47	2	1 cement + 1% Intrusion Aid	0.03	0.64 0.50 0.41 0.36 0.34	45 33 24 16 15	100 100 100 100 100	40 46 40 45 46	27 32 21 24 24	15 17 21 25 24	0 0 2 2 2	---	
54	44	2	1 cement + 1% Intrusion Aid	0.02	1.00 0.67 0.55 0.46 0.41 0.40	24 14 8 5 3 2	50 50 50 50 50 50	32 36 36 36 33 33	24 25 24 24 23 23	15 15 14 14 14 13	0 0 0 0 2 2	---	
55	38	2	1 cement + 1% Intrusion Aid	0.03	0.60 0.52 0.46 0.41 0.39 0.37 0.35	36 24 15 9 7 5 3	50 50 50 50 50 50 50	37 35 36 40 36 36 34	26 25 25 21 24 25 23	16 14 14 14 14 15 15	0 0 0 0 0 1 2	---	
56	38-A	2	1 cement + 1% Intrusion Aid + 1.5% sand passing No. 50 sieve	0.03	0.50	--	50	--	--	--	--	---	Sand plugged specimen
57	46-A	2	1 cement + 1% Intrusion Aid	0.03	0.45	--	50	--	--	--	--	---	Shear test specimen
58	31	2	1 cement + 1% Intrusion Aid	0.01	4.0	0	25	0	0	0	0	---	Refused grout
59	32	2	1 cement + 1% Intrusion Aid	0.02	1.50 1.00 0.75 0.60 0.50 0.43	9 6 4 2 1 1	25 25 25 25 25 41	17 17 17 17 17 32	14 14 13 13 13 23	9 9 8 9 9 30	0 0 0 0 0 20	---	Frothy grout. Pressure increased and static pressure maintained on specimen approx 10 min after pumping
60	33	2	1 cement + 1% Intrusion Aid	0.03	1.00 0.75 0.60 0.46 0.40 0.38 0.36	25 18 12 6 4 2 2	25 25 25 25 25 25 25	17 18 18 20 19 18 18	12 12 15 14 14 14 14	7 7 7 9 5 8 9	0 0 0 0 0 0 0	---	Grout from test 33 used in test 33-A

(Continued)

* Consistency measured on torque meter with spider wire diameter increased to 0.5625 in.

Table 1 (Continued)

Serial No.	Test No.	Stage No.	Grout	Crack Thick-ness in.	Water-cement Ratio (by Wt)	Flow cu ft/hr	Pressure (psi) Gage No.					Consistency (Torque Deg)	Remarks
							0(A)	1	2	3	4(B)		
61	33-A	2	1 cement + 1% Intrusion Aid	0.03	0.36	2	25	19	14	5	0	---	Crack in specimen. Turned vertically, long axis horizontal
62	65	2	1 cement + 0.153% RDA	0.01	2.00 1.38 1.06 0.95	7 5 2 0	100 100 100 ---	80 80 82 --	57 58 64 --	46 48 60 --	0 0 0 --	Grout passed through No. 100 sieve	
63	62	2	1 cement + 0.153% RDA	0.02	1.20 0.75 0.60 0.55 0.50 0.46 0.43 0.40	37 26 18 15 11 8 6 4	100 100 100 100 100 100 100 100	75 66 68 71 68 66 68 68	47 42 46 46 45 43 43 45	27 25 26 26 26 25 26 26	0 0 0 0 0 0 0 0	---	
64	63	2	1 cement + 0.153% RDA	0.03	1.20 0.86 0.67 0.54 0.50 0.43 0.40	64 44 40 26 20 14 11	75 42 60 100 100 100 100	56 32 41 70 75 73 73	40 21 27 45 50 46 44	25 13 16 27 27 26 26	0 0 0 0 0 0 0	Could not get pressure up to 100 psi at first	
65	67	2	1 cement + 0.153% RDA	0.03	0.83 0.62 0.50 0.38 0.36 0.33	55 49 44 36 25 7	42 62 52 100 100 100	33 40 52 52 56 44	24 29 40 40 40 31	17 20 27 28 28 19	0 0 0 0 4 2	Specimen split longitudinally, test void	
66	51	2	1 cement + 0.153% RDA	0.01	2.00	--	50	--	--	--	--	---	Test void, specimen leaked at shims
67	51	2	1 cement + 0.153% RDA	0.02	0.46 0.43 0.41 0.40 0.36	8 6 5 4 2	50 50 50 50 50	38 38 36 36 36	25 24 24 24 24	18 15 13 13 13	1 1 1 1 2	---	
68	60	2	1 cement + 0.153% RDA	0.02	2.00 1.00 0.75 0.60 0.50 0.47	31 24 16 10 6 4	50 50 50 50 50 50	38 37 36 37 38 38	25 24 25 25 25 25	15 15 15 15 15 15	0 0 0 0 0 0	Static pressure maintained approx 10 min	
69	61	2	1 cement + 0.153% RDA	0.03	1.00 0.86 0.67 0.55 0.46 0.43 0.40	50 50 50 50 50 50 50	33 34 34 35 36 35 50	22 23 23 23 22 21 50	13 14 13 12 12 12 50	0 0 0 0 0 0 0	---	Static pressure maintained approx 10 min	
70	79	2	1 cement + 0.20% RDA	0.03	1.00 0.71 0.55 0.45 0.42 0.38 0.36	43 36 24 14 11 7 3	50 50 50 50 50 50 50	32 32 32 32 32 36 47	40 35 35 36 38 39 50	12 12 11 11 12 12 36	0 0 0 0 0 0 0	10* 21 45 55 135 217 356	Static pressure maintained approx 10 min
71	56	2	1 cement + 0.20% RDA	0.01	2.00 1.43 1.11	6 4 0	25 25 25	-- -- --	-- -- --	0 0 0	0 0 0	---	
72	57	2	1 cement + 0.20% RDA	0.02	1.11 0.91 0.77 0.67 0.63 0.56 0.50 0.45	14 12 6 5 5 4 2 1	25 25 25 25 19 19 19 25	18 19 13 13 18 19 13 25	13 13 7 7 13 13 7 13	7 7 7 7 7 7 7 20	0 0 0 0 0 0 0 0	---	Used grout from test 56. Static pressure maintained approx 10 min
73	58	2	1 cement + 20% RDA	0.03	1.00 0.83 0.71 0.63 0.56 0.50 0.45 0.43	32 26 20 15 10 7 4 3	25 25 25 25 19 19 19 25	19 18 18 19 19 19 19 25	14 13 13 13 13 13 13 25	8 7 7 7 7 7 7 20	0 0 0 0 0 0 0 0	---	Static pressure maintained approx 10 min
74	12	1	1 cement:1 fly ash + 1% Intrusion Aid	0.01	4.30	0	100	--	--	--	0	---	Roughened surfaces on specimen. Refused grout

(Continued)

* Consistency measured on torque meter with spider wire diameter increased to 0.5625 in.

Table 1 (Continued)

Serial No.	Test No.	Stage No.	Grout	Cement:water thickness (by wt.)	Flow (in. per hr.)	Pressure (psi)						Consistency (Torque in.)*	Remarks
						100	200	300	400	500	600		
74	1	1	1 cement:1 fly ash + 1% Intrusion Aid	0.01	4.38	0	100	--	--	--	0	--	Refused grout
76	8	1	1 cement:1 fly ash + 1% Intrusion Aid	0.02	1.19	0	100	--	--	--	0	--	Roughened surfaces on specimen. Refused grout
77	23	1	1 cement:1 fly ash + 1% Intrusion Aid	0.02	1.27	16	55	--	--	--	0	--	Static pressure maintained approx 10 min
				1.00	18	90	--	--	--	0	--		
				0.82	17	100	--	--	--	0	--		
				0.70	16	100	--	--	--	0	--		
				0.61	14	80	--	--	--	0	--		
				0.51	10	80	--	--	--	0	--		
				0.45	7	80	--	--	--	0	--		
				0.44	6	90	--	--	--	0	--		
				0.42	5	100	--	--	--	0	--		
				0.40	4	100	--	--	--	0	--		
				0	100	--	--	--	--	0	--		
78	5	1	1 cement:1 fly ash + 1% Intrusion Aid	0.03	0.35	24	50	--	--	--	0	0	Roughened surfaces on specimen
				0.57	15	58	--	--	--	0	0		
				0.53	13	60	--	--	--	0	0		
				0.49	6	96	--	--	--	0	0		
				0.45	5	100	--	--	--	0	0		
				0.43	0	100	--	--	--	0	0		
79	19	1	1 cement:1 fly ash + 1% Intrusion Aid	0.03	0.44	24	100	--	--	--	0	--	
				0.41	17	100	--	--	--	0	0		
				0.37	18	100	--	--	--	0	0		
				0.35	7	100	--	--	--	0	0		
				0.33	0	100	--	--	--	0	0		
80	45-A	2	1 cement:1 fly ash + 1% Intrusion Aid	0.02	0.40	2	50	36	19	12	0	--	Aid added to grout from test 45. Grout thickened to 0.38 water-cement ratio, did not refuse, grout oily looking
				0.38	1	50	35	19	12	0	0	--	
81	40	2	1 cement:1 fly ash + 1% Intrusion Aid	0.03	0.50	17	50	34	33	12	0	--	
				0.44	7	50	20	14	7	0	0	--	
				0.40	6	50	27	18	10	1	0	--	
				0.37	3	50	27	20	11	2	0	--	
82	40-A	2	1 cement:1 fly ash + 1% Intrusion Aid	0.03	0.50	--	50	--	--	--	--	--	Test inconclusive. Fly ash dry sieved through No. 100 sieve before using
83	50	2	1 cement:1 fly ash + 1% Intrusion Aid	0.03	0.45	--	50	--	--	--	--	--	Grout not thickened to refusal, shear test specimen. Pressure maintained 15 min after pumping
84	41	2	1 cement:1 fly ash + 1% Intrusion Aid	0.01	3.00	--	25	0	0	0	0	--	Refused grout
85	42	2	1 cement:1 fly ash + 1% Intrusion Aid	0.02	3.00	34	25	17	12	6	0	--	
				1.50	14	25	16	7	6	0	0	--	
				1.49	8	25	18	12	7	0	0	--	
				0.96	0	25	22	14	5	0	0	--	
86	43	2	1 cement:1 fly ash + 1% Intrusion Aid	0.03	0.81	23	25	17	12	6	0	--	
				0.77	13	25	17	11	6	0	0	--	
				0.64	7	25	17	12	6	0	0	--	
				0.46	2	25	17	12	6	0	0	--	
				0.43	0	30	22	14	5	0	0	--	
87	74	2	1 cement:1 fly ash + 1% RDA	0.02	1.00	17	100	28	16	8	0	--	
				0.75	12	100	20	12	6	0	0	--	
				0.57	6	100	23	14	7	0	0	--	
				0.50	3	100	23	11	6	0	0	--	
88	68	2	1 cement:1 fly ash + 0.70% RDA	0.03	0.71	67	50	62	43	25	2	--	Specimen cracked near entrance end for grout. Grout film thickness may have exceeded 0.03 in.
				0.56	47	100	74	48	27	2	0	--	
				0.50	42	100	80	55	30	2	0	--	
				0.45	34	100	80	55	31	2	0	--	
				0.41	20	100	80	51	28	0	0	--	
				0.38	14	100	77	51	26	0	0	--	
				0.35	8	100	78	51	26	3	0	--	
89	73	2	1 cement:1 fly ash + 0.70% RDA	0.03	1.00	64	100	70	52	32	0	--	
				0.75	55	100	70	45	30	0	0	--	
				0.60	37	100	50	35	21	0	0	--	
				0.50	24	100	48	33	19	0	0	--	
				0.43	17	100	66	48	28	0	0	--	
				0.40	14	100	68	48	27	0	0	--	
				0.37	8	100	66	46	26	0	0	--	
				0.35	4	100	55	40	23	0	0	--	
90	56	2	1 cement:1 fly ash + 0.70% RDA	0.02	3.00	26	50	27	21	14	0	--	Static pressure approx 10 min after pumping
				1.00	71	50	31	21	13	0	0	--	
				0.60	50	35	24	14	0	0	0	--	
				0.50	6	50	36	25	15	0	0	--	
				0.43	3	50	36	25	15	0	0	--	
				0.40	2	50	38	27	16	0	0	--	
				0.38	1	50	44	30	15	0	0	--	
				0	30	39	35	17	0	0	0	--	

(Continued)

* Consistency measured on torque meter (piano-wire viscometer) with spider wire of 0.125-in. diameter

Table 1 (Continued)

Serial No.	Test No.	Stage No.	Grout	Crack Thick-ness in.	Water-cement Ratio (by Wt)	Flow cu ft/hr	Pressure (psi) Gage No.				Consistency (Torque Deg)	Remarks
							0(A)	1	2	3		
91	53	2	1 cement:1 fly ash + 0.20% Marasperse "C"	0.02	3.00	29	50	33	23	15	0	---
					1.00	23	50	34	24	14	0	---
					0.60	11	50	32	22	13	0	---
					0.50	7	50	34	23	15	0	---
					0.43	5	50	35	23	15	0	---
					0.40	3	50	36	23	15	0	---
					0.38	2	50	36	23	15	0	---
					0.35	1	50	--	23	15	0	---
						50	--	50	47	0		
												Marasperse "C" brand calcium lignosulfonate used instead of RDA in this test. Static pressure for approx 10 min
92	71	2	1 cement:1 fly ash + 0.20% RDA	0.03	1.00	56	50	36	25	15	0	---
					0.75	38	50	36	26	15	0	---
					0.60	27	50	37	26	14	0	---
					0.50	13	50	38	26	15	0	---
					0.45	9	50	41	29	16	0	---
					0.43	4	50	37	28	15	0	---
					0.40	1	50	41	29	16	0	---
						50	48	41	29	13	0	---
93	80	3	1 cement:1 fly ash + 0.20% RDA	0.03	1.00	59	50	32	40	15	0	9*
					0.71	46	50	26	38	12	0	15
					0.55	17	50	13	23	4	0	30
					0.45	3	50	13	25	5	0	51
					0.42	4	50	9	23	2	0	71
					0.38	3	50	11	24	4	0	92
94	69	2	1 cement:1 fly ash + 0.20% RDA	0.02	1.50	16	25	25	15	7	0	---
					1.00	11	25	23	15	8	0	---
					0.75	8	25	22	15	7	0	---
					0.67	6	25	23	16	8	0	---
					0.60	5	25	23	16	8	0	---
					0.56	4	25	23	16	8	0	---
					0.50	2	25	22	16	8	0	---
					0.45	2	25	23	15	8	0	---
					0.43	0	25	--	--	0	0	---
						25	23	16	19	15	0	
95	70	2	1 cement:1 fly ash + 0.20% RDA	0.03	1.00	35	25	17	12	6	0	---
					0.75	23	25	16	10	5	0	---
					0.67	20	25	17	11	5	0	---
					0.60	15	25	17	11	5	0	---
					0.56	11	25	17	11	6	0	---
					0.50	8	25	17	11	6	0	---
					0.45	4	25	17	11	6	0	---
					0.43	2	25	17	11	6	0	---
						25	26	25	23	13	0	---
96	81	3	1.5 cement:1 fly ash + 0.20% RDA	0.03	1.00	58	50	34	27	14	0	9*
					0.71	50	50	31	23	11	0	16
					0.55	33	50	30	23	11	0	30
					0.45	20	50	32	24	11	0	62
					0.42	14	50	33	26	12	0	85
					0.38	8	50	26	21	10	0	122
					0.36	6	50	29	24	11	0	145
97	82	3	2.0 cement:1 fly ash + 0.20% RDA	0.03	1.00	58	50	26	20	19	0	9*
					0.71	44	50	24	16	17	0	16
					0.55	30	50	26	20	18	0	37
					0.45	17	50	30	21	18	0	79
					0.42	12	50	26	20	18	0	116
					0.38	8	50	26	20	18	0	208
					0.36	2	50	18	16	18	0	354
						50	17	26	24	0	0	---
Static pressure for approx 10 min												

Note: Test 1 saved surfaces on specimen. Tests 2 through 12, surfaces of specimens lightly rubbed by carborundum stone. All other tests conducted with specimen surfaces smooth, as cast against plate glass.

* Consistency measured on torque meter with spider wire diameter increased to 0.5625 in.

Table 2
Quality of Hardened Grout Films from Appearance

Grout	Crack Thickness, in.		Crack Thickness, in.				
	0.01	0.02	0.03	0.01	0.02	0.03	
<u>100-PSI Pumping Pressure</u>							
Neat	Water-cement ratio 1.34. Incompletely filled crack; poor-quality, porous, ridged film. Bond badly. Segregated. Bond to top slab poor.	Water-cement ratio 0.56. Crack filled with fair-quality grout. Some segregation and bleeding. Bond to top slab poor.	Water-cement ratios 0.32 and 0.36. Crack filled with good-quality grout. No evidence of bleeding or segregation. Bond to top slab good.	1 cement:1 slag	No test	No test	Water-cement ratio 0.47. Very good-looking grout film. No bleeding evident. Bond to top slab poor.
1 cement:1 fly ash	Water-cement ratio 1.05. Filled with hard grout. Segregated, channelled. Fair-quality film, nonsegregated, nonbleeding grout. Bond to top slab good.	Water-cement ratios 0.49 and 0.44. Filled with good-quality, hard, nonsegregated, nonbleeding grout. Bond to top slab good.	1.5 cement:1 slag	No test	No test	No test	Water-cement ratio 0.47. Filled with good-looking grout film. Very little bleeding. Bond to top slab poor.
1 cement:1 fly ash + 1% Intrusion Aid	Water-cement ratio 1.30. Partially filled with poor-quality, stringy, bleeding grout. Gel voids. Bond to top slab poor.	Water-cement ratio 0.40. Fairly well-filled with fair-quality, moderately bleeding grout. Bond to top slab poor.	2 cement:1 slag	No test	No test	No test	Water-cement ratio 0.47. Filled with good-looking grout. No bleeding. Gas voids evident. Bond to top slab poor.
1 cement:1 fly ash + RDA	No test	Water-cement ratio 0.50. Filled with poor-quality grout. Considerable bleeding. Bond to top slab poor.	1 cement:1 fly ash + RDA	No test	Water-cement ratio 0.35 and 0.38. Filled with good-quality grout. No bleeding. Bond to top slab poor.	Water-cement ratio 0.38. Good quality grout. Slight bleeding. Bond to top slab poor.	Water-cement ratio 0.47. Filled with good-quality grout. Bond to top slab good.
Neat	Water-cement ratio 1.33. Crack filled with porous, channelled, ridged film. Bond excessively segregated. Bond to top slab poor.	Water-cement ratio 0.48. Crack filled with fair-quality film. Bond to top slab poor.	Water-cement ratios 0.46 and 0.38. Filled with good-quality grout. Bond to top slab fair.	1.5 cement:1 fly ash + RDA	No test	No test	Water-cement ratio 0.47. Good-quality grout. Slight bleeding. Bond to top slab good.
1 cement:1 fly ash	No test	Water-cement ratio 0.40. Water-cement ratio 0.38. Water-cement ratio 0.45. Water-cement ratio 0.45.	Water-cement ratios 0.38 and 0.45. Bond to top slab poor in both cases.	2 cement:1 fly ash + RDA	No test	No test	Water-cement ratio 0.47. Filled with good-quality grout. Bond to top slab poor.
1.5 cement:1 fly ash	No test	No test	Neat	Water-cement ratio 2.57.	Water-cement ratio 0.46.	Water-cement ratio 0.45. Water-cement ratio 0.45.	Water-cement ratio 0.45. Well-filled with good-quality grout. Bond to top slab poor.
2 cement:1 fly ash	No test	No test	1 cement:1 fly ash + 1% Intrusion Aid	Water-cement ratio 4.0. Incomplete filling with watery, stringy grout.	Water-cement ratio 0.52.	Water-cement ratio 0.46. Water-cement ratio 0.46.	Water-cement ratio 0.45. Filled with good-quality grout. Bond to top slab poor.

25-PSI Pumping Pressure

Grout	Crack Thickness, in.		Crack Thickness, in.				
	0.01	0.02	0.03	0.01	0.02	0.03	
<u>25-PSI Pumping Pressure</u>							
Neat	Water-cement ratio 1.34. Crack filled with hard grout. Bond to top slab poor.	Water-cement ratio 0.56. Crack filled with good-quality grout. Bond to top slab poor.	Water-cement ratio 0.45. Hard ridges, bleeding evident. Bond to top slab poor.	1 cement:1 fly ash + 1% Intrusion Aid	No test	No test	Water-cement ratio 0.45. Filled with fair-quality grout. Considerable bleeding. Bond to top slab poor.
1 cement:1 fly ash	No test	No test	Water-cement ratio 3.00. Partially filled with small clumps of poor-quality grout. Bond to top slab poor.	Water-cement ratio 0.45. Filled with hard but stringy grout. Much bleeding. Bond to top slab fair.	No test	No test	Water-cement ratio 0.45. Well-filled with good-quality grout. Bond to top slab poor.

Table 3

Consistency and Bleeding of Third-stage Grouts

Grout	Water-cement Ratio 0.4				Water-cement Ratio 0.6*			
	ASTM C-243		ASTM C-243		ASTM C-243		ASTM C-243	
	Rate $\text{cm}^3/\text{cm}^2/\text{sec}$	Capacity cm^3/cm^3	Rate $\text{cm}^3/\text{cm}^2/\text{sec}$	Capacity cm^3/cm^3	Rate $\text{cm}^3/\text{cm}^2/\text{sec}$	Capacity cm^3/cm^3	Rate $\text{cm}^3/\text{cm}^2/\text{sec}$	Capacity cm^3/cm^3
	$\times 10^6$	$\times 10^3$						
Neat cement	350	80	37	37	38	19	38	19
1 cement:1 fly ash	332	65	38	45	45	17	45	17
2 cement:1 fly ash	337	65	38	6	20	10	20	10
Cement + RDA	374	50	50	20	20	17	20	17
1 cement:1 fly ash + RDA	258	55	36	36	36	12	36	12
2 cement:1 fly ash + RDA	263	55	36	36	36	12	36	12
1 cement:1 slag	378	73	28	28	28	23	28	23
2 cement:1 slag	355	69	33	33	33	26	33	26
1 cement:2 pumicite	**	53	18	18	18	32	18	32
2 cement:1 pumicite	**	40	14	14	14	24	14	24
1 cement:1 opaline shale		Too dry to test	787	787	787	86	787	86
2 cement:1 opaline shale		Too dry to test	244	244	244	38	244	38

* Fluid grouts could not be made at 0.4 water-cement ratio using the opaline shale. Ratio of 0.6 tested for information only; this ratio represents about lowest ratio for fluid grout with this material.

** Exceeded capacity of meter.

Table 4
Results of Leaching Tests on Grouts

Specimen	Grout Age Days	Water-cement Ratio 0.4						Water-cement Ratio 0.7					
		Total Sol			Ca ion			Total Sol			Ca ion		
		pH	ppm	%	ppm	%	pH	ppm	%	ppm	%	ppm	%
<u>Neat Cement</u>													
Flask 1	9	12.0	920	0.26	121	0.08	12.0	1354	0.59	180	0.17		
	14	11.7	650	0.18	189	0.12	11.5	772	0.34	234	0.23		
	35	11.6	888	0.25	170	0.11	11.6	752	0.33	177	0.17		
	97	11.3*	702	0.20	33	0.02	11.2*	202	0.09	22	0.02		
Flask 2	92	11.5*	880	0.25	103	0.06	11.7*	1278	0.56	84	0.08		
	97	11.4	628	0.18	130	0.11	11.2	176	0.08	48	0.05		
	118	11.5	766	0.22	165	0.10	11.1	152	0.07	15	0.01		
	180	11.6	838	0.24	146	0.09	11.5	82	0.39	191	0.18		
<u>Cement + RDA</u>													
Flask 1	9	11.7	756	0.22	150	0.09	11.8	1192	0.57	137	0.14		
	14	11.6	712	0.20	193	0.12	11.6	900	0.43	193	0.20		
	35	11.6	1030	0.30	189	0.12	11.6	966	0.46	159	0.17		
	97	11.3*	430	0.12	29	0.02	11.6	840	0.40	188	0.20		
Flask 2	92	11.4	648	0.19	100	0.06	11.5	1008	0.48	75	0.09		
	97	11.4	690	0.20	167	0.11	11.6	902	0.43	156	0.16		
	118	11.6*	876	0.25	156	0.10	11.6	920	0.44	170	0.19		
	180	11.6	968	0.28	113	0.07	11.7	1022	0.49	226	0.23		
<u>1 Cement:1 Fly Ash</u>													
Flask 1	9	11.5	794	0.23	75	0.08	11.6	1246	0.57	104	0.17		
	14	11.5	634	0.19	126	0.14	11.6	780	0.36	172	0.29		
	35	10.9**	130	0.04	12	0.01	11.3	654	0.30	95	0.16		
	97	10.2	202	0.00	7	0.01	11.2*	382	0.17	13	0.02		
Flask 2	92				Discarded		11.3*	680	0.31	13	0.02		
	97				Discarded		10.9	156	0.07	26	0.04		
	118				Discarded		10.9	126	0.06	18	0.03		
	180				Discarded		10.4	188	0.09	13	0.02		
<u>2 Cement:1 Fly Ash</u>													
Flask 1	9	11.4	810	0.23	112	0.09	11.6	1524	0.69	149	0.20		
	14	11.4	620	0.18	157	0.13	11.6	660	0.30	170	0.23		
	35	11.4**	614	0.18	118	0.10	11.4**	670	0.30	110	0.15		
	97	10.2	70	0.02	11	0.01	11.3	470	0.21	54	0.07		
Flask 2	92	11.2*	302	0.09	43	0.04	11.3*	940	0.42	48	0.06		
	97	11.0	120	0.03	33	0.03	10.0	20	0.01	11	0.01		
	118	11.1	214	0.06	36	0.03	10.0	34	0.02	8	0.01		
	180	10.9	322	0.09	34	0.03	9.9	56	0.03	9	0.01		
<u>1 Cement:1 Fly Ash + RDA</u>													
Flask 1	9	11.7	824	0.24	82	0.09	11.8	1374	0.63	78	0.13		
	14	11.5	626	0.19	104	0.11	11.5	760	0.35	115	0.19		
	35	11.3**	530	0.16	32	0.04	11.5	93	0.42	63	0.11		
	97	10.5	134	0.04	7	0.01	11.3	738	0.34	9	0.02		
Flask 2	92	10.9*	156	0.05	11	0.01	11.3*	1644	0.75	9	0.02		
	97	10.8	124	0.04	24	0.03	10.5	130	0.06	13	0.02		
	118	10.6	144	0.04	8	0.01	10.6	148	0.07	12	0.02		
	180	10.5	162	0.05	6	0.01	10.5	140	0.06	8	0.01		
<u>2 Cement:1 Fly Ash + RDA</u>													
Flask 1	9	11.6	954	0.28	93	0.08	11.7	1526	0.70	23	0.13		
	14	11.5	654	0.19	142	0.12	11.6	840	0.38	151	0.20		
	35	11.4	728	0.21	92	0.08	11.5	902	0.41	84	0.11		
	97	11.3	464	0.14	7	0.01	11.3	552	0.25	10	0.01		
Flask 2	92	11.3*	606	0.18	21	0.01	11.5*	996	0.45	20	0.03		
	97	11.0	184	0.05	42	0.04	11.2	204	0.09	36	0.05		
	118	11.0	148	0.04	31	0.03	10.6	88	0.04	11	0.01		
	180	10.9	142	0.04	9	0.01	10.2	22	0.04	5	0.01		

(Continued)

* Flasks broken prior to test age.

** Flasks cracked prior to test age.

Table 4 (Continued.)

Specimen	Grout Age Days	Water-cement Ratio 0.7						Water-cement Ratio 0.8					
		pH	Total Sol ppm	Ca ion ppm	Ca ion %			pH	Total Sol ppm	Ca ion ppm	Ca ion %		
<u>1 Cement:1 Slag</u>													
Flask 1	9	11.5	856	0.25	56	0.01		11.7	1124	0.50	107	0.13	
	14	11.3	592	0.17	129	0.10		11.3	662	0.30	130	0.16	
	35	11.1*	348	0.10	22	0.02		11.3*	510	0.23	54	0.07	
	97	10.5	224	0.07	7	0.01		10.5	210	0.09	9	0.01	
Flask 2	92	10.6*	98	0.03	11	0.01		11.5*	560	0.39	17	0.02	
	97	11.3	248	0.07	48	0.04		11.5	354	0.16	105	0.13	
	118	10.7	160	0.05	13	0.01		10.2	114	0.05	9	0.01	
	180	10.4	242	0.07	12	0.01		10.4	462	0.21	10	0.01	
<u>2 Cement:1 Slag</u>													
Flask 1	9	11.4	724	0.21	32	0.07		11.6	1220	0.54	131	0.15	
	14	11.4	600	0.17	139	0.10		11.5	776	0.35	162	0.18	
	35	9.6*	200	0.06	7	0.01		11.4	1026	0.46	128	0.14	
	97	10.6	212	0.06	7	0.01		11.3*	332	0.17	15	0.02	
Flask 2	92	11.5*	722	0.21	33	0.07		11.7*	1444	0.64	124	0.14	
	97	11.5	306	0.09	112	0.08		11.5	466	0.21	141	0.16	
	118	11.1	180	0.05	28	0.02		10.6	92	0.04	18	0.02	
	180	11.1	192	0.06	20	0.01		11.3	300	0.13	45	0.05	
<u>1 Cement:1 Pumicite</u>													
Flask 1	9	11.8	1076	0.34	167	0.21		11.7	1008	0.46	109	0.19	
	14	11.7	976	0.31	182	0.23		11.6	844	0.39	150	0.26	
	35	11.4	914	0.29	78	0.10		11.4	762	0.35	76	0.13	
	97	11.1*	280	0.09	9	0.01		10.9*	230	0.13	7	0.01	
Flask 2	92	11.4	770	0.24	90	0.11		11.3*	414	0.19	52	0.09	
	97	11.4	562	0.18	132	0.17		11.3	396	0.14	89	0.16	
	118	11.3	402	0.13	93	0.12		11.2	188	0.09	27	0.05	
	180	11.3	330	0.10	32	0.04		10.9	736	0.11	10	0.02	
<u>1 Cement:1 Pumicite</u>													
Flask 1	9	11.7	964	0.76	156	0.14		11.8	1220	0.55	134	0.21	
	14	11.5	734	0.47	184	0.16		11.5	872	0.39	204	0.27	
	35	11.1	1134	0.75	134	0.12		11.6	940	0.42	136	0.18	
	97	10.7*	1134	0.05	5	0.01		11.1*	334	0.15	9	0.01	
Flask 2	92	11.4*	746	0.17	147	0.13		11.5*	326	0.42	106	0.14	
	97	10.7	70	0.02	10	0.01		10.0	32	0.01	8	0.01	
	118	11.4	616	0.18	132	0.12		11.4	570	0.26	152	0.20	
	180	11.1	36	0.01	10	0.01		11.4	390	0.18	40	0.05	
<u>1 Cement:1 Opaline Shale</u>													
Flask 1	9	11.4	632	0.24	97	0.14		11.4	616	0.28	91	0.16	
	14	11.5	512	0.20	115	0.17		11.4	592	0.27	100	0.18	
	35	11.3	344	0.13	56	0.08		11.3	380	0.17	45	0.08	
	97	10.7	90	0.03	17	0.02		10.6	146	0.07	17	0.03	
Flask 2	92	11.3	260	0.10	61	0.03		11.0*	176	0.08	9	0.02	
	97	11.1	124	0.05	44	0.06		10.5	96	0.04	14	0.02	
	118	10.9	104	0.04	37	0.05		10.5	98	0.04	16	0.03	
	180	10.7	128	0.05	38	0.04		10.1	78	0.04	14	0.02	
<u>2 Cement:1 Opaline Shale</u>													
Flask 1	9	11.3	776	0.31	133	0.16		11.4	700	0.41	97	0.13	
	14	11.5	572	0.23	134	0.16		11.5	716	0.32	128	0.17	
	35	8.5*	34	0.01	6	0.01		11.4	502	0.23	122	0.17	
	97	11.0	146	0.06	3	0.01		10.7*	130	0.05	19	0.02	
Flask 2	92	11.3	426	0.17	130	0.16		11.0*	154	0.07	18	0.02	
	97	11.3	374	0.13	111	0.15		11.5	280	0.13	91	0.12	
	118	11.3	330	0.09	76	0.09		10.2	132	0.06	13	0.02	
	180	9.3*	54	0.02	21	0.03		11.0	142	0.06	17	0.02	

* Flasks broken prior to test age.

APPENDIX A: TESTS ON SIMULATED GROUTS AND
EXAMINATION OF MATERIAL FILLING
FISSURE IN TEST 9, STAGE 1

Test Conditions

1. During the Stage 1 tests in order to determine bleeding and setting-time characteristics of grouts of several water-cement ratios and to determine the characteristics of a high water-cement ratio grout, such as that required to grout the specimen of test 9, certain tests described in detail in this appendix were made. Samples of each type of grout used in Stage 1 of the program were made with different water ratios as follows:

<u>Grout Mixture No.</u>	<u>Water-cement Ratio, wt</u>
<u>Neat Grout</u>	
1	3.81
2	2.06
3	0.33
3A	1.93
<u>1 Cement:1 Fly Ash</u>	
4	1.01
5	0.44
<u>1 Cement:1 Fly Ash + 1% Intrusion Aid</u>	
6	4.32
7	0.36

These grouts, except 3A, were observed for time of set and were checked to determine the approximate amount of bleeding that occurred. On mixture 3A, only the procedure given below was followed:

- a. The mixture was agitated after mixing for 2 hr and 35 min.
- b. One hundred milliliters of grout was placed in a 150-ml vial, stoppered, and stored in a vibration-free, constant-temperature room for periodic observation of cement-water interface.
- c. A portion was placed in a shallow glass dish to a depth of 1/2 in., the dish was covered and placed in the fog room (73.4 \pm 2 F) for examination after 13 days.
- d. A portion was placed between glass plates shimmed 0.01 in. apart, the edges sealed, and the plates placed in the fog room for examination at 13 days.

e. Additional portions were placed in four small stoppered vials, stored in the fog room, and examined at 1, 3, 7, and 13 days.

Time of Set

2. The time-of-set tests were made using the Vicat time-of-set needle (1-mm diameter) with the grout contained in 130-ml, wide-mouthed bottles approximately 1.75 in. in diameter by 3.06 in. high. Measurements were made for depth of penetration below the cement-water interface. The grout conditions tested and setting times obtained are shown in the following tabulation. The setting-time results are plotted on fig. A1.

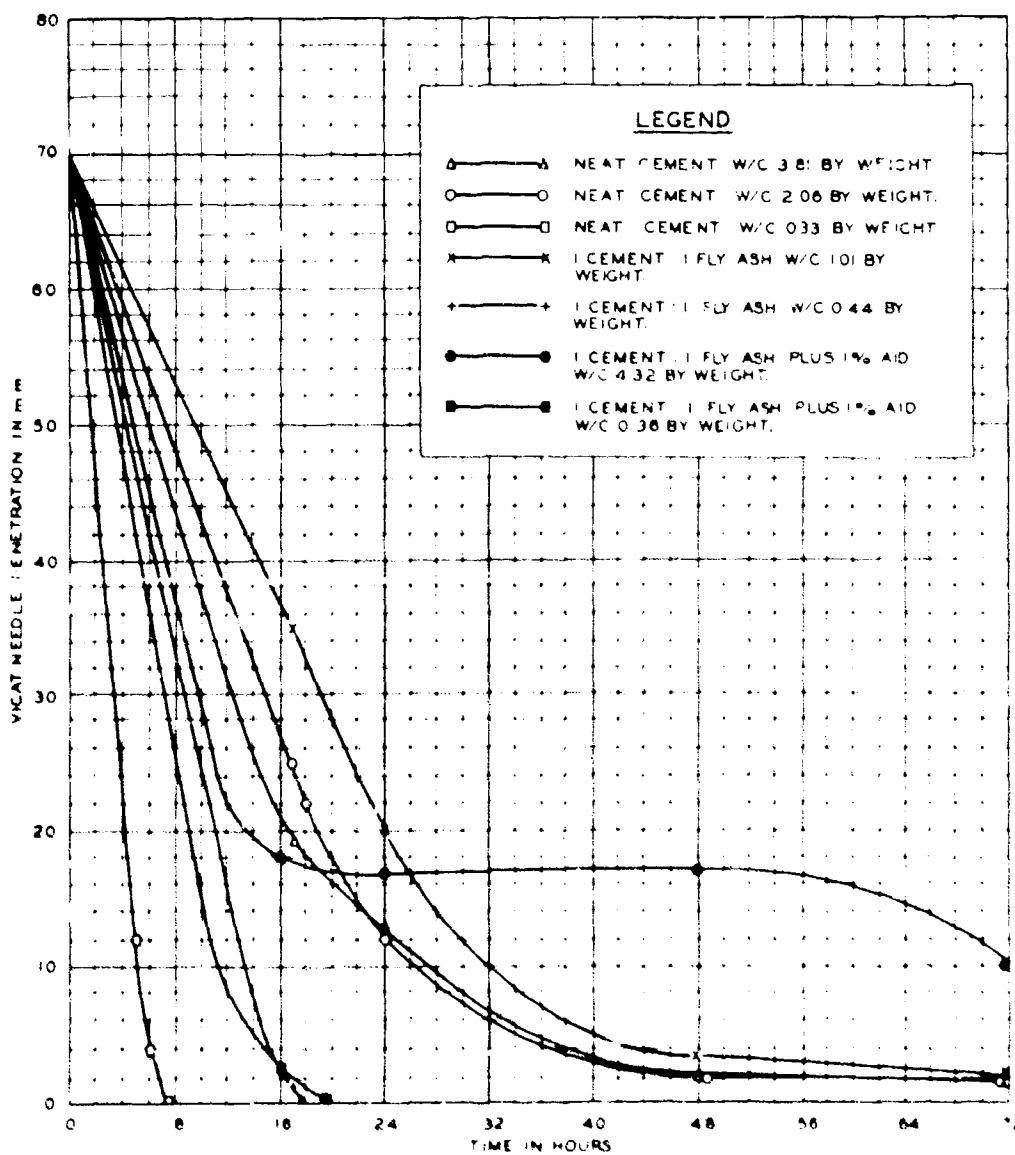


Fig. A1. Setting time of grouts

Mixture No.	Water-cement Ratio wt	Time of Set hr
<u>Neat Grout</u>		
1	3.81	233
2	2.06	233
3	0.33	7.25
<u>1 Cement:1 Fly Ash</u>		
4	1.01	16.5
5	0.44	16.5
<u>1 Cement:1 Fly Ash + 1/2 Intrusion Aid</u>		
6	4.32	*
7	0.36	20

* Not set at 336 hr (14 days). Penetration 4 mm.

Mixtures 1, 2, and 4 developed a thin white film on top which allowed penetration for many hours. This film was raked off one surface with the operator's finger but the needle still penetrated slightly until final set was reached. The penetration values on fig. A1 are averages of penetrations of mixtures with and without supernatant water removed. Removal of the water did not seem to have any noticeable effect on time of set. The surface of the paste in mixture 6 was crusted over and then crumbled, making it difficult to determine just where the top of the uncrumbled paste really was.

3. The setting times were found to be roughly proportional to the water contents of the grouts being tested; however, the two thinnest neat grouts, water-cement ratios of 3.81 and 2.06, both had setting times of 233 hr, almost 10 days, and the cement-fly ash-Intrusion Aid grout, with a water-cement ratio of 4.32, had not set at the end of 14 days. It is believed that the setting time would also be influenced to some degree by the thinness of the grout film, i.e., the thinner the film the longer the setting time, since the cement grains would not be able to settle out and form the relatively constant water-cement concentration as is the case in thick films, or in grouts placed in relatively deep containers.

Bleeding

4. Tests for bleeding were made by placing 500 ml of grout on a 1000-ml graduate and observing the amount of clear water that had separated after one hour. Results are shown in the tabulation below.

Mixture No.	Water-cement Ratio wt	Bleed Water ml	Bleeding %
<u>Neat Grout</u>			
1	3.81	93	71
2	2.06	62	48
3	0.33	0	0
<u>1 Cement:1 Fly Ash</u>			
4	1.01	22	17
5	0.44	0	0
<u>1 Cement:1 Fly Ash + 1% Intrusion Aid</u>			
6	4.32	90	69
7	0.36	0	0

5. Fig. 22 of the main report indicates that the bleeding experienced with neat grout having a water-cement ratio of 3.81 was about 87 per cent. The above tabulation lists the bleeding as being 71 per cent. Both values, even though they do not agree, are extremely high and indicate what occurs when sedimentation takes place in grout with this high water content. Bleeding was 48 per cent at a water-cement ratio of 2.06. This value conforms very closely to the curve of fig. 22. The bleeding was 0 per cent with a water-cement ratio of 0.33, which also agrees with the curve. With the cement-fly ash grout and a water-cement ratio of 1.01, the bleeding was 17 per cent in this series, and approximately 23 per cent on the curve. With a water-cement ratio of 0.44, the bleeding was 0 per cent in both the tabulation and curve. The bleeding percentage of 69 for the cement-fly ash-Intrusion Aid grout with a water-cement ratio of 4.32 has no counterpart on the curve; however, from the shape of the curve of the Intrusion grout on fig. 22 the percentage of bleeding at this water content would be expected to be less for the Aid-bearing

grout than for neat grout. No bleeding was indicated either in the tabulation or on the curve for cement-fly ash Intrusion Aid grout at a water-cement ratio of 0.36.

Petrographic Examination of Specimens
of Grout Mixture 3A at 13 Days

6. The graduate, mold, and shimmed glass plates were inspected before they were opened. The supernatant water in the graduate was clear, but a thick film of white to grayish scum floated on the surface of the water. A large number of small water-clear crystals were attached to the inner wall of the graduate between the cement-water interface and the surface of the water. Similar crystals could be seen on the upper surface of the grout.

7. The shimmed glass plates were examined through the upper plate, using a stereoscopic microscope. There were many crystals adhering to the upper plate, in interfering masses. In some, the broad flat hexagonal shape of $\text{Ca}(\text{OH})_2$ was clearly visible. Some air bubbles had been caught between the plates in the preparation of the specimen. The tops of the bubbles were separated from the upper glass by a thin film of solid material which did not contain any crystals large enough to be recognized with a stereoscopic microscope. Some subsidence or shrinkage of the grout film appeared to have taken place. When the shimmed glass plates were separated, the grout film stuck to the lower plate, and the hydroxide crystals to the upper. The grout could be scratched with a fingernail, was coherent, and looked like an aggregate of fine grains of silt size. The Pyrex mold was tilted while being moved from the fog room to the stereoscopic microscope room, and loose material on top of the grout was agitated so that the supernatant water was slightly turbid. Some crystals of calcium hydroxide could be seen on the upper surface of the grout.

8. The supernatant water in the small sealed vial had a thick film in the meniscus, and hydroxide crystals were adhering to the glass and lying on the upper surface of the grout. Since the vial appeared similar

to the graduate and the Pyrex mold, it was decided that a petrographic examination would be made of the sealed vial, and a chemical analysis of the other specimens.

Results of Examination of Bleed Water and Paste

9. The results of the examinations at 1, 3, 8, and 13 days are reported in table A1, and can be summarized as follows:

- a. At 1, 3, 8, and 13 days the unhydrated portions of the cement, calcium hydroxide, and calcium carbonate were the only crystalline constituents detected in the bleed water and the paste.
- b. At all ages, amorphous gel formed by hydration could be found as rims around unhydrated cores. The gel carbonated very quickly when air had had access, developing small spherulites and incomplete spherulites of calcium carbonate.
- c. Only one crystal form of calcium hydroxide was found; a hexagonal plate, bounded by the hexagonal prism (or two trigonal prisms) and basal pinacoids (fig. A2). Groups

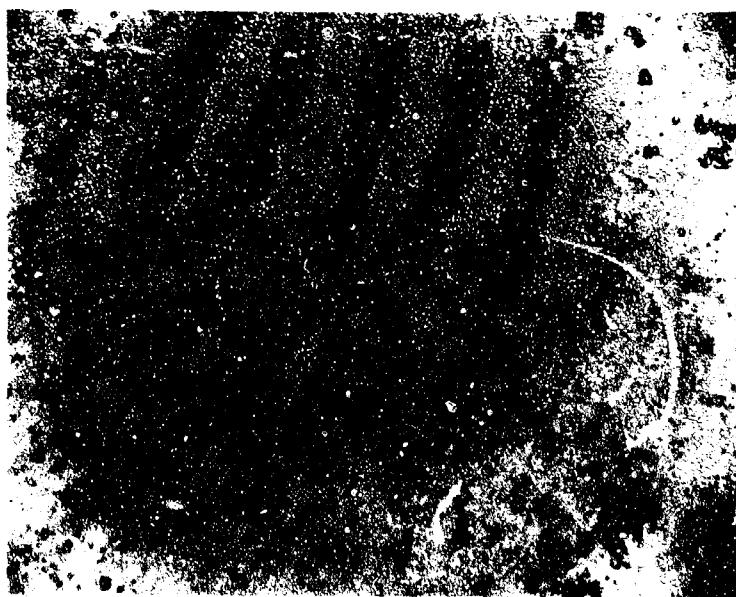


Fig. A2. Calcium hydroxide crystal from bleed water, X110, plane light. A thin film of carbonating gel overlies and surrounds the crystal, and clumps of carbonating gel and unhydrated cement are scattered through the field. The diameter of the crystal as measured in the A-B direction was 0.20 mm. The crystal has the forms common in calcium hydroxide - the pinacoid and prism

formed by the intergrowth of several plates increased in size, number, and complexity with increasing age. The size of the single crystals also increased with age. Some of the single crystals and some of the members of the groups showed variations in extinction position; a few of the interference figures obtained were biaxial with a very small optic angle but more were uniaxial negative. Where the diameter and thickness of the same crystal could be measured, the diameter was about 10 times the thickness on crystals about 0.2-0.3 mm in maximum diameter.

- d. No calcium sulfoaluminate was found in any of the freshly prepared immersion mounts at 1, 3, 8, or 13 days. The high sulfate form was found in unsealed slides of bleed water from the 13-day vial, after the slides had been allowed to evaporate for several days. The high sulfate form occurred as prisms and needles with parallel extinction, negative elongation, and indices of refraction slightly above and slightly below 1.4645 (sodium light). Radial groups of needles with parallel extinction and positive elongation were also present. Some had indices of refraction slightly above and below 1.50. Others, in two other slides, were similar but had one index of refraction slightly above 1.485 and below 1.489, and the other slightly below 1.485. Both agree better in optical properties with dehydrated high sulfate calcium sulfoaluminate than with any other compound likely to be present in bleed water. The analyses of bleed water reported in paragraph 4 indicate that such small amounts of SO_3 and Al_2O_3 were present in the solution that no high sulfate calcium sulfoaluminate would be expected to crystallize from unconcentrated bleed water.
- e. The secondary calcium carbonate found was most abundant in the scum in the meniscuses, and was principally in the form of spherulites and incomplete spherulites of vaterite A (calcite with interstitial water). This particular modification of calcium carbonate forms in moderately alkaline solutions.
- f. There was a decrease in unhydrated cement grains with increasing age.
- g. Several of the slides of residue from evaporated bleed water contained an unidentified crystalline material in elongated blades with slightly inclined extinction, which was interpreted as deformed sulfoaluminate. Subsequent evidence, described below, suggests that this interpretation was wrong.

Results of Examination of Thin Sections

10. Thin sections of the 1- and 3-day hydrates contained calcium

hydroxide crystals in random orientations, large amounts of partially hydrated and unhydrated cement, and an amorphous groundmass of gel. The general outlines of the calcium hydroxide were usually apparent and usually indicated single crystals rather than groups. There appeared to be a well-developed parting in the hydroxide normal to the crystallographic c axis; extinction was parallel to the parting, which is usually apparent even where the outlines of the grain are irregular.

11. Several changes were apparent in the 30-day as compared to the 3-day hydrates. Parts of the groundmass appeared to be crowded with extremely small units of crystalline material, which were not clearly resolved at a magnification of 900 diameters. The units were very small relative to the size of the large calcium hydroxide crystals, and were somewhat lower in interference colors, which suggests that they are calcium hydroxide, rather than calcium carbonate. The large calcium hydroxide crystals had increased in relative size, and frequently included several partly hydrated grains of cement. Calcium hydroxide also appeared as narrow rims around unhydrated cement grains. All five thin sections of the 30-day hydrate contained a crystalline compound not observed in the 1- and 3-day sections, which resembled the unidentified crystalline compound found in the residue of evaporated bleed water. It formed elongated crystals and radiating groups, with parallel extinction in some and slightly inclined extinction in others, negative elongation, and birefringence somewhat greater than that of calcium hydroxide. These properties do not agree with reported properties of any common constituent of cement hydrate, with crystalline hydrates described in the available literature, or with those of gypsum, plaster of Paris, or anhydrite.

Petrographic Examination of Grout
Filling Fissure in Test 9

12. The grout proportions used in this test (see table 1 of main report) were the same as in mixture 3A. The specimen was placed in the fog room, after grouting, and was opened and examined at an age of 13 days.

13. It was observed upon inspection, shortly after the specimen was opened, that virtually none of the cementing material had adhered to or recently been in contact with the upper surface of the fissure. The upper surface was incrusted with a white deposit which appeared to be composed to a large extent of small but visible crystals, the individual faces of which were of sufficient size to give reflections visible to the naked eye. Examination of this upper surface with a stereoscopic microscope indicated an essentially continuous coating of finely divided white material upon which had grown an abundance of well-developed hexagonal crystals. These crystals were markedly tabular in habit with a diameter estimated to be approximately ten times the thickness. The majority of the crystals grew on the surface so oriented that the attachment was along one edge of the table, otherwise there appeared to be no preferred orientation. Among the areas incrusted with crystals were paths or channels which appeared to represent places along which fluid had moved. Fewer crystals appeared to have developed along these channels, and in some cases the coating of finely divided white material in the channels was observed to contain a typical network of shrinkage cracks probably caused by evaporation after the specimen was opened. An examination of selected crystals and determination of optical constants revealed that the crystals had the properties of calcium hydroxide and are therefore presumed to be derived from the cement.

14. An examination of the lower surface of the fissure shortly after the specimen had been opened revealed an almost continuous layer of wet unhardened siltlike substance. In small areas, this material was darker in color and appeared to have hardened, particularly in the immediate vicinity of the point of inflow and outlet. Over the remaining area the general softness of the matter indicated an essentially complete absence of set. A rather poorly developed pattern of flow lines was noted. The siltlike deposit when scraped from a small area with a knife blade and reworked slightly, developed a marked water sheen indicating a high free-water content. The material showed no tendency toward plastic behavior when rubbed between the fingers, but felt more like silt. Additional observations, approximately 48 hr after opening, revealed that the

deposit had dried out considerably and had become almost completely white in color. The change in color is regarded as principally a function of drying, although it is believed to be the result, in part, of alteration of the calcium hydroxide to calcium carbonate by reaction with the carbon dioxide in the air. The wet siltlike substance, previously described, had dried out but showed no signs of having set although in drying it had caked to some extent.

15. Examination of the siltlike material by petrographic means included observation of the fact that it was largely soluble with effervescence in cold dilute hydrochloric acid, suggesting that it contained a considerable amount of gel that had carbonated prior to examination. The residue after acid treatment appeared to consist of gel with very small particles of a crystalline substance.

16. All the petrographic findings applicable to the simulated grout 3A appear to apply fully to the filling material in test 9.

Chemical Tests on Simulated Grout Mixture 3A

Tests

17. Supernatant water and sludge from the graduate, pie plate, and shimmmed plate were analyzed chemically. The results of these tests are shown in the following tabulation.

Source of Sludge	Free Water %	Percentages (Calculated on a Dry Basis)									
		Ign. Loss	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Total	Na ₂ O	K ₂ O
Graduate	41.1	13.2	19.8	4.0	3.4	55.3	2.4	1.4	99.5	.06	.22
Mold	46.6	13.3	19.8	3.8	3.4	55.6	2.3	1.5	99.7	.06	.28
Shimmmed plate	23.5*	13.2	19.9	3.9	3.4	55.5	2.4	1.1	99.4	.06	.34

* Low value due largely to evaporation during examination of the open plate under stereoscopic microscope.

18. Ignition loss of the sludge probably represents bound water, chiefly, and not CO₂. The ignition loss of cement RC-183 was 0.60. Recalculating the above percentages using an ignition loss of 0.60 the following percentages are obtained.

All

Source of Sludge	Percentage									
	Ign. Loss	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Total	Na ₂ O	K ₂ O
Graduate	0.6	22.8	4.6	3.9	63.7	2.7	1.6	99.9	0.06	0.22
Mold	0.5	22.8	4.4	3.9	64.0	2.6	1.7	100.0	0.06	0.28
Shimmed plate	0.6	22.9	4.5	3.9	63.9	2.7	1.2	99.7	0.06	0.34
RC-183 cement	0.6	22.8	4.3	3.8	63.2	3.1	1.5	99.5	0.17	0.48

19. The ignition loss of the sludge in the tabulation in paragraph 18 is perhaps too high, since the Na₂O and K₂O values are lower than the values for the RC-183 cement. All results agree closely, the greatest variation being in the CaO, MgO, Na₂O, and K₂O. An alkali content of the sludge lower than that of the original cement is expected, since the supernatant water contained a high percentage of Na₂O and K₂O. The filtrate from the silica determination was used to determine the sodium and potassium content using the Beckman flame spectrophotometer. The results are therefore probably too high because of interferences by other elements and compounds present (Ca, Mg, SO₃, etc.).

Source of Water	Parts per Million								
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	Total
Graduate (5-ml portion)	110	0	0	770	0	89	248	1184	2401
Mold (20-ml portion)	15	0	0	532	2	2	558	2496	3603

20. Only small aliquots of water were analyzed: therefore, any small error would be magnified by dilution factors. The top surfaces of the cement pastes were rather soft with the remainder of the pastes grainy and hard, but not as hard as the usual cement pastes become upon standing. Perhaps some of the solids on the surface of the sludge were obtained in pipetting the water. This would account for an increase in values. The analysis was recalculated as percentages of dissolved solids:

Source of Water	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	Total
Graduate	4.6	0	0	32.1	0	3.7	10.3	49.3	100.0
Mold	0.4	0	0	14.7	0	0.1	15.5	69.3	100.0

21. All results were determined gravimetrically except values of

Na_2O and K_2O , which were determined on the Beckman flame spectrophotometer. The water level in the 150-ml graduate dropped a total of 2.2 ml in 13 days; the cement-water interface settled 43 ml during the first hour and remained constant thereafter.

Summary of chemical tests
of simulated grout mixture 3A

22. Less free water was found in the grout from the shimmed plate than in that from the other specimens, probably as a result of evaporation from the plate during examination. The free water in the grout from the graduate was less than that in the Pyrex mold; the contact surface between grout and bleed water was relatively greater in the mold. There was close agreement among the three specimens in ignition loss (bound water) and in oxide determinations. Recalculation of the results using the ignition loss found in the analysis of the cement indicated relatively large decreases in Na_2O and K_2O in the grout as compared with the cement. Relatively high concentrations of Na_2O and K_2O were found in the bleed water, with CaO , SiO_2 , and SO_3 . The amounts of Fe_2O_3 , Al_2O_3 , and MgO found in the bleed water did not differ significantly from zero. The cement paste in the graduate settled to its final level after one hour, and the water level in the graduate decreased 2.2 ml in 13 days.

Chemical Examination of Sludge
from Fissure of Test 9

23. A portion of the material filling the fissure in test 9 was taken for chemical analysis, the results of which provide the following comparative data between the siltlike matter and the cement.

	<u>Silt</u>	<u>Cement RC-183</u>
SiO_2 , %	23.2*	22.8
Al_2O_3 , %	10.9	8.1
(Al_2O_3 , %)	6.6	4.3)
(Fe_2O_3 , %)	4.3	3.8)
(Continued)		

* Average of two determinations (22.8 and 23.5).

	<u>Silt</u>	<u>Cement RC-183</u>
MgO, %	2.9	3.3
CaO, %	58.3	63.2
Remainder, %	4.6*	2.6

* Since the analysis is on an ignited basis, this is not H₂O nor CO₂.

It is apparent from these data that the siltlike material had been somewhat leached of lime -- as would be expected from the fact that a deposit of calcium hydroxide was found on the upper surface of the fissure.

Table A1

Observations of Contents of Sealed Vials of Cement Paste with Supernatant Liquid, Stored in Fog Room, at Four Ages

Meniscus

1 day

Floating scum; contains flat hexagonal plates of hydroxide to which some isotropic, easily carbonated material and a few grains of unhydrated cement adhere. Measured hydroxide crystals range from 0.04 to 0.15 mm in maximum diameter, with the more common values 0.04-0.07 mm.

3 days

Floating scum; resembles that from 1-day vial but the size of the hydroxide ranges up to 0.19 mm. The scum also contains gel which carbonates rapidly and a small amount of unhydrated cement.

8 days

Thick scum; average size of hydroxide greater than in 3-day scum; otherwise similar to 1- and 3-day specimens.

13 days

Thick scum; average size of hydroxide crystals considerably larger than at 8 days; range from 0.29 to 0.39 mm; otherwise similar to 1- and 3-day specimens.

Supernatant Liquid

1 day

Clear, rare hydroxide crystals and small amount of easily carbonated amorphous material.

3 days

Clear; rare hydroxide crystals and small amounts of easily carbonated amorphous material.

8 days

Clear; scattered shining hydroxide crystals adhering to wall of vial and lying on paste. Hydroxide crystals up to 0.37-mm maximum diameter.

13 days

Clear; abundant shining hydroxide crystals adhering to walls and lying on top of paste.

(Continued)

Table A1 (Continued)

Paste

1 day

Dissecting needle pressed by finger penetrates about 3/4 in. Paste looks like silt-stone; is coherent but easily broken in the hand; many deformed hydroxide plates growing parallel to the glass on the vial walls and the outside of the plug. Under petrographic microscope cement grains with unhydrated cores are most abundant constituent; some adhere to each other; some have gel rims. Hydroxide crystals are commonly single, but a few groups were found. Single crystals range in maximum dimension from 0.06 to 0.13 mm; 0.13 mm most common value.

3 days

Dissecting needle pressed by finger penetrates about 3/4 in. Similar to 1-day paste under stereoscopic microscope and under petrographic microscope; gel rims are broader than in 1-day hydrate, and groups of intergrown hydroxide crystals are more common. Size ranges from individual crystals 0.03 mm to groups 0.25 mm in maximum diameter.

8 days

Paste similar to 3-day paste under stereoscopic microscope. Single hydroxide crystals up to 0.42 mm across; their thickness is about 1/10 of their maximum diameter. The hydroxide groups are more complex than those in the 3-day paste; gel is more abundant but there is still a great number of recognizable cement grains.

13 days

Dissecting needle only penetrates about 3/8-1/2 in. Single hydroxide crystals up to 0.51 mm in maximum diameter; abundant gel and recognizable cement grains. Hydroxide groups larger and more complex than in 8-day paste.

APPENDIX B: CONSIDERATIONS INVOLVED IN STRENGTH AND
POROSITY OF HIGH RATIO WATER-CEMENT GROUTSStrength

1. It is suggested that the phenomenon exhibited by the material filling specimen 9 represents the behavior of a cement-water mixture in which the water-cement ratio is so extremely high that the resulting product has properties more like those of laitance than cement paste. It is also believed that the absence of contact between the siltlike material deposited on the lower surface and the upper surface indicates that considerable sedimentation occurred prior to the growth of the calcium hydroxide crystals since the crystals give every indication of having grown out from the upper face into the space that was filled with material that offered no restraint to the crystal growth. It is estimated that the siltlike deposit did not fill much more than half the thickness of the fissure. It is suggested that the water-cement ratio of the substance deposited on the lower surface of the fissure was so high that the space between cement grains, which in normal pastes and concretes is of capillary proportions and subsequently becomes largely filled with gel, was so great that it was not generally possible for the gel developed by the hydration of each individual particle or group of particles to grow out through the space and make sufficient contact with that developing from other particles or groups of particles. If such were the case the ratio of the volume of gel to the volume of available space would be very small. As has been pointed out by Powers and Brownyard,* the strength that develops by the setting and hardening of portland-cement pastes is a function of this ratio. Bogue** says the cement glue or gel appears to be an amorphous colloid. During the process of hydration the volume of the solid phase increases, that of the liquid water decreases, but the

* "Physical properties of hardened portland cement paste," Journal, American Concrete Institute, vol 43, pp 845-857.

** The Chemistry of Portland Cement, p 467.

volume of the system, cement plus water, decreases. Selective settling of the larger or heavier grains takes place in the early stages of the reaction, before a stiffening of the paste, leaving a layer of fine material with a high water content known as laitance.

2. The following is based on the work of Powers and Brownyard*:

a. The increase in the bulk volume of a paste upon hydration is:

$$0.860 (w_n + 4V_m)$$

where

w_n = nonevaporable water

V_m = weight of water proportional to the gel material in the paste ($4V_m$ = weight of water required to fill all the voids in the gel)

0.860 = mean specific volume of evaporable and non-evaporable water.

b. Hence the ratio of the increase in solid phase to the original space available is:

$$\frac{0.860 (w_n + 4V_m)}{w_o}$$

where

w_o = original water content of paste

$\frac{V_m}{V_c}$ = a constant.

c. Therefore the ratio of increase in solid phase to original water content (X^1) is:

$$X^1 = 0.860 \frac{V_m}{w_o} \frac{(1 + 4k)}{(k)}$$

d. The compressive strength (f_c) is related to X^1 as follows:

$$f_c = m X^1 + B$$

* Op. cit., p B1.

where

$$B = \text{interception } f_c$$

$$m = \text{slope.}$$

e. If

$$m^1 = 0.860 \frac{(1 + 4k)}{(k)} m$$

then

$$f_c = m^1 \frac{V}{w_o} + B .$$

f. For observed values of f_c for 4 cements the constants found are:

$$f_c = 120,000 \frac{V}{w_o} - 3600 .$$

g. Thus compressive strength is shown to be directly proportional to the increase in $\frac{V}{w_o}$ regardless of age, original water-cement ratio, or identity of the cement.

h. From pp 596-597 (Powers and Brownyard)

$$V_m = 0.02 \text{ to } 0.05 \text{ g per g original cement or on the average } 0.04 \text{ lb per lb dry cement.}$$

3. From the equation in paragraph 2 f it is seen that if

$\frac{V}{w_o} = \frac{1}{33}$, the compressive strength will be zero. If $V_m = 0.04$ g per g

dry cement, then for 83.3 lb of cement $V_m = 3.3$. If it is assumed that by sedimentation all of the cement particles settle to form a deposit in the lower half of the fissure leaving the upper half filled with free water: then

$$w_o = 161.1 \times 1/2 = 80.5$$

and

$$\frac{V}{w_o} = \frac{3.3}{80.5} = \frac{1}{24.2} ,$$

or the same order of magnitude as $\frac{1}{33}$; hence the conditions of no compressive strength may be explained. These calculations are offered purely as a suggestion regarding the order of magnitude of the relationship since the values 120,000 and 3,600 were derived for the constants in this equation from a study of the compressive strength of 2-in. cubes made with four cements only and are not necessarily applicable to conditions such as those under consideration at this time.

Net Water Content and Porosity of the Cement Paste

4. The water-cement ratio in the paste after bleeding is calculated to be 0.74 (or 8.35 gal per bag), based on the information obtained by observations of water-level and cement-water interface in the graduate. The porosity of the hardened paste may be calculated for a volume of 46 ml of paste in the graduate after subsidence using the formulas given by Powers and Brownyard,* the data on the grout mixture and volumes in the graduate, and the values found for ignition loss of the paste.

Total porosity = volume of gel pores plus volume of capillary pores (1)

$$\text{Volume of gel pores} = 3.6 \times K W_n = 3.6 \times 0.26 \times 10.0 = 9.4 \text{ ml} \quad (2)$$

where

K = a constant depending on the compound composition of the cement; for this cement, = 0.26, and

W_n = nonevaporable water in paste, taken as ignition loss of paste and calculated to ml, = 10.0.

$$\begin{aligned} \text{Volume of capillary pores} &= W_o = 0.86 (1 + 4K) W_n = 32.2 - 19.0 \\ &= 14.7 \text{ ml} \quad (3) \end{aligned}$$

* "Physical properties of hardened portland cement paste," Journal, American Concrete Institute, vol 18, No. 8, part 9, pp 973-976.

where

W_o = original water content after bleeding, g or ml
= 32.2, and K and W_n as in equation 2.

The total porosity in 46 ml of paste is therefore

$$9.4 + 14.7 = 24.1, \text{ or 52 per cent by volume.}$$

5. The permeability of pastes of relatively high water-cement ratio is probably determined largely by the capillaries outside the gel. The capillary space in this paste amounts to about 32 per cent by volume.